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INDEX

General Page

Low-Temperature Properties of Structural Materials: Compilation of Published Data 54
Low-Temperature Properties of Ferrous Materials 54
Chemical Milling 55

Nickel

Theory of Ferromagnetism of Nickel 55
Embrittlement of High-Purity Nickel 55
Cleaning Techniques in the Electronics Industry 56
Study of Laves Phases and AB_5 Compounds Among Transition Elements 56

Electrodeposition and Other Coating Methods

Levelling Action During Nickel and Copper Plating 56
Influence of Plating Temperature on the Properties of Bright-Nickel Deposits 57
Solution Transfer in Barrel Plating 57
Application of Statistical Quality Control to Plating Processes 57
Adhesion of Nickel on Substrates Contaminated with Fatty Acids 58
Adhesion of Organic Enamels to Electrodeposited Nickel 58
Corrosion and Electrode-Potential Studies of Nickel/Chromium Coatings 58
Corrosion-Resistance of Nickel/Chromium-plated Zinc Die Castings 59
Experience with the CASS Test 59
Deposition and Performance of Duplex-Chromium Coatings 60
Corrosion Characteristics of Chromium Overcoatings 60
Electroless Deposition of Nickel-Phosphorus Alloys from Pyrophosphate Solutions 60
Electrodeposition and Magnetic Properties of Cobalt-Nickel Alloys 61
Deposition of Cobalt-Nickel Alloys from Sulphamate Solutions 61
Faraday's Laws Applied to Cleaning of Basis Metals 62

Non-Ferrous Alloys

Report of the Royal Mint: Nickel-containing Coinage 62
Yield-Point Phenomena in Copper- and Nickel-base Alloys 62
Yield-Point and Order-Hardening Phenomena in Nickel Silvers 62

Cast Iron

Properties of Alloy Cast Irons 63
Properties of Spheroidal-Graphite Alloy Cast Irons 63
Hot-Hardness of Steels and Spheroidal-Graphite Cast Iron 64

Constructional Steels

Vacuum-Induction Melting of High-Strength Steels 64
Size Effects in Notched-Bend Tests on Low-Alloy Steels 65
Influence of Cold Work on the Structure and High-Temperature Strength of Steel 65

Constructional Steels (*contd.*) Page

Low-Temperature Flow and Fracture Characteristics of Low-Alloy Steel 65
Low-Temperature Tests on Pressure-Vessels Fabricated from 9 per cent. Nickel Steel 66
Influence of Rapid Tempering on Susceptibility to 500°F. Embrittlement 67
Determination of Hydrogen-Embrittlement Effects in Cadmium-Plated High-Strength Steel 68
Influence of Nickel Coatings on the Susceptibility of Cadmium-Plated Steel to Hydrogen Embrittlement 68
Non-Embrittling Nitrate-containing Cadmium-Plating Solution 69
Response of Low-Alloy Steels to the ARDE Smoothing Process 69

Heat- and Corrosion-Resisting Materials

Determination of Proof Stress, Rupture Strength and Creep Strength at Elevated Temperatures: British Standards 69
Development of Improved Nickel-base High-Temperature Alloys 70
Fatigue Data on Precipitation-Hardenable Stainless Steels 70
Metal Wear by Scoring 70
Factors Influencing Corrosion in Furnace Atmospheres 71
Anodic Passivity in Metals 71
Cathodic Protection of Metals 71
Thermodynamics of Corrosion in Fused Chlorides 71
Intergranular Oxidation of 18-8 Stainless Steels by Oxygen and Dry Sodium Chloride 71
Bibliography on Stress Corrosion 72
Stress-Corrosion Cracking of Austenitic Chromium-Nickel Stainless Steels: A.S.T.M. Report 72
Comparative Stress-Corrosion Behaviour of Various Grades of Stainless Steel 74
Feasibility of Preventing Stress Corrosion by Establishing Threshold Values 75
Influence of Surface Coatings on the Susceptibility of Steels to Stress Corrosion 75
Caustic Stress Corrosion of 18-12-Nb Steel 75
Stress-Corrosion Cracking of Stainless Steel De-aerator Trays 76
Corrosion-Resistance of Cast Nickel-Molybdenum-Iron Alloys 76
Influence of Moisture on the Resistance of Materials to Corrosion by Bromine 77
Influence of Moisture on the Resistance of Materials to Chlorohydrocarbons 77
Design and Operation of Ethanolamine Gas-Scrubbing Systems 78
Corrosion-Erosion of Sensitized Stainless Steel in a ThO_2/UO_3 Slurry 78
Resistance of Nickel-containing Materials to Liquid Fluorine and Liquid Oxygen 79
Low-Temperature Properties of Cold-Worked or Welded Stainless-Steel Sheet 79
Preferential Corrosion of Stabilized Stainless-Steel Welds 79
Combined Method for Analysis of High-Alloy Irons and Steels 80

Analysis 80

ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Low-Temperature Mechanical Properties of Structural Materials: Compilation of Published Data

R. M. McCLINTOCK and H. P. GIBSON: 'Mechanical Properties of Structural Materials at Low Temperatures: A Compilation from the Literature.'

U.S. Dept. of Commerce, National Bureau of Standards, Monograph 13, June 1, 1960; 180 pp.

In this compilation of data available from published and, in some cases, unpublished literature (the monograph contains a *bibliography* of 104 items), the tensile strength, yield strength, tensile elongation and impact energy of approximately 200 materials are presented graphically as functions of temperatures in the range 4°–300°K. The authors have compiled the data 'with the idea that an engineer who is making initial calculations on equipment for operation at cryogenic temperatures is more interested in obtaining quickly a definite figure than he is in evaluating the experimental data given in several detailed reports on the same material. The graphs and tables presented here consequently represent an attempt by the authors to perform an evaluation of data which have appeared in the literature, and to present the design engineer with the result. The curves therefore appear as lines representing the mechanical properties as functions of temperature, and not as bands representing maximum and minimum values reported.'

The classes of material covered by the publication are indicated below.

Aluminium and Its Alloys.

Copper and Its Alloys [brasses and bronzes (including nickel-aluminium bronze), cupro-nickels, nickel silvers].

Nickel and Its Alloys ('Inconel', 'Monel', 'K Monel', 'H Monel', 'S Monel', nickel-chromium resistance alloys).

Titanium and Its Alloys.

Magnesium and Its Alloys.

Austenitic Stainless Steels (A.I.S.I. Types 201, 202, 301, 302, 303, 304, 308, 310, 316, 321, 347; cast grades).

Ferritic and Hardenable Stainless Steels (A.I.S.I. Types 400, 410, 430; 'AM-350', 'F.V. 520B', '17-4 P.H.' and '17-7 P.H.' precipitation-hardenable steels).

Low-Alloy Constructional Steels (including ship-plate steels, '300M', 'Hy-Tuf', 'T-1', 9 per cent. nickel, and various A.I.S.I. nickel or nickel-containing grades).

'*Superalloys*' ('A-286', 'D-979', 'AF-183', 'N-155', 'Haynes Stellite 25', '19-9DL', 'V-36', 'S-816', 'Hastelloy A', 'Hastelloy B', 'Hastelloy C' and 'Hastelloy X').

Brazing and Soldering Metals.

Miscellaneous Materials (including manganese austenitic steels, high-expansion steel, nickel-containing flake-graphite and S.G. irons, various nickel-iron alloys).

Non-Metallic Materials (plastics and glass).

Low-Temperature Properties of Ferrous Materials

SOC. AUTOMOTIVE ENGINEERS: 'Low-Temperature Properties of Ferrous Materials.'

Information Report TR-169, revised [May 1959; 9 pp.

This review of information available on the influence of low-temperature on the properties of ferrous materials is in nine sections. Throughout the report the points raised in the text are supplemented by data derived from the literature, and reference is made to a *bibliography* of 47 items. The heads and sub-heads employed give an adequate idea of the scope of the review and of the individual sections, and are therefore reproduced below.

General Effect of Low Temperature.
Notch-Toughness Behaviour (transition temperature; stress and strain rate).
Notch-Toughness Evaluation (impact tests; tests involving welding; drop-weight and explosion tests).
Correlation Among Test Results.
Evaluation of Service Performance.
Influence of Welding.
Influence of Design and Fabrication Procedures.
Influence of Composition and Microstructure (hot-rolled and normalized steel; carbon, phosphorus and nitrogen; manganese; silicon and aluminium; sulphur; chromium, copper, vanadium, nickel; molybdenum; ferrite grain size; hardened steels;

tempered martensite; embrittlement in quenched-and-tempered steels; stainless steels; malleable and nodular irons).
Notch-Toughness Needed in Service.

Chemical Milling

C. C. SHEPHERD: 'Chemical Milling.'
47th Ann. Tech. Proc., Amer. Electroplaters' Soc., 1960, pp. 51–4; disc., pp. 229–30.

Application of the chemical-milling process to materials difficult to machine by conventional methods has increased rapidly in recent years. Although the process was initially restricted to aluminium, it is now being successfully used on magnesium, steel, titanium and molybdenum, and materials once considered impossible to mill in acid solutions (such as the ultra-high-strength low-alloy steels) are being satisfactorily processed.

In this paper the author draws attention to the applications for which chemical milling has been found suitable, discusses the main stages involved in the process (preparation, masking, etching and stripping), and briefly considers the effects of milling on the properties of the component being fabricated.

NICKEL

Theory of Ferromagnetism of Nickel

J. SEIDEN: 'Theory of Ferromagnetism of Nickel.'
Comptes Rendus, 1960, vol. 251, Nov. 21, pp. 2311–13.

In a recent paper (*ibid.*, 1960, vol. 251, p. 1062), the author sought to explain ferromagnetism of metals in terms of a generalized Heisenberg model. In the present note, he shows that this model is satisfactorily applicable to the magnetic properties of nickel at low temperature.

Embrittlement of High-Purity Nickel

K. M. OLSEN, C. F. LARKIN and P. H. SCHMITT: 'Embrittlement of High-Purity Nickel.'
Amer. Soc. Metals, 1960, *Preprint* 203.
Trans. Amer. Soc. Metals, 1961, vol. 53, pp. 349–58.

It has been shown that a sulphur content as little as 0·01 wt. per cent. is sufficient to cause fracture in nickel during forging (hot and cold malleability is satisfactory, however, at sulphur levels of 0·005 per cent. or less). In a comprehensive study of the influence of minor constituents on the emission and life characteristics of oxide-coated nickel cathodes, OLSEN found that malleability was good in the presence of small single additions of silicon, aluminium, carbon, boron, cobalt, zirconium, tungsten, titanium, manganese and magnesium. During a study of the influence of these additions on the tensile properties, electrical resistance and recrystallization characteristics of nickel (see abstract in *Nickel Bulletin*, 1959, vol. 32, No. 6, p. 171), an unusual embrittling behaviour associated with recrystallization was observed, an observation which was unexpected in view of the good forgeability of the nickel. The present paper describes an exploratory investigation of the causes of the embrittlement.

Recrystallization curves were obtained from tensile and resistivity measurements made on 0·025-in.-diameter (0·625-mm.-diameter) wire which had been cold-drawn 70 per cent. and heat-treated for ½ hour in dry hydrogen at various temperatures. The wire was obtained from different ingots of high-purity nickel.

From the data presented it is concluded that grain-boundary embrittlement associated with segregation of a nickel-sulphide phase can occur in cold-worked high-purity nickel containing as little as 0·0009 wt. per cent. sulphur, but only as a result of heat-treatment in the recrystallization grain-growth range 450°–600°C. Embrittlement is not induced if the same cold-worked nickel is heat-treated at the usual 800°C. annealing temperature, and it is postulated that the sulphide phase is precipitated early in the recovery-recrystallization stage and is carried along in the boundaries with further grain growth. At the higher annealing temperatures, the sulphide is believed to diffuse back into the nickel matrix, thereby restoring ductility.

Contamination of the high-purity nickel resulted from pick-up of sulphur from the magnesium-oxide crucible during melting, but it was found that virtually sulphur-free nickel could be prepared using a crucible which had been conditioned by a 'wash' melt of nickel to absorb and remove the available sulphur from the crucible.

Addition of 0·01 per cent. oxygen did not eliminate the brittleness induced by recrystallization of high-purity nickel containing about 0·001 per cent. sulphur. A 0·03 per cent. addition of manganese had only a slight effect on brittleness, but introduction of 0·025 per cent. magnesium completely eliminated the embrittling behaviour.

No embrittlement occurred in pure nickel, nickel+0·01 per cent. oxygen, or nickel+0·03 per cent. manganese, when the sulphur concentration was 0·0005 per cent. or less, and, indeed, all these materials exhibited exceptionally good ductility after heat-treatment in the 400°–600°C. range. The improvement in ductility associated with lowering the concentration of sulphur is deemed important, since the increase in malleability will facilitate fabrication by such cold-working operations as press forming, stamping, tube drawing and strip rolling.

Cleaning Techniques in the Electronics Industry

D. E. KOONTZ, D. O. FEDER and C. O. THOMAS: 'Cleaning in the Electronics Industry.'

47th Ann. Tech. Proc., Amer. Electroplaters' Soc., 1960, pp. 188–98; disc., pp. 252–3.

Trace amounts of extraneous materials may significantly influence either the electrical or fabrication characteristics of modern electronic devices. In this paper, the authors allude to trends in the electronics industry which have created a need for more effective control over both the type and amount of residual contaminant. Contaminants are then classified, and their effects on the performance of typical devices (e.g., nickel electron-tube components) are exemplified. Practical techniques developed for the detection, removal and control of contaminants are outlined, and the integration of cleaning techniques into typical cleaning cycles of interest to electroplaters is discussed.

Low-Temperature Mechanical Properties of Structural Materials: Compilation of Published Data

See abstract on p. 54.

Study of Laves Phases and AB_5 Compounds Among Transition Elements

A. E. DWIGHT: 'Factors Controlling the Occurrence of Laves Phases and AB_5 Compounds Among Transition Elements.'

Amer. Soc. Metals, 1960, *Preprint No.* 211.
Trans. Amer. Soc. Metals, 1961, vol. 53, pp. 477–99.

The author gives details of a study carried out to obtain information on the interrelations of atomic size, stacking geometry and electronic structure in the occurrence of the three Laves phases of AB_2 composition and two compounds of AB_5 composition (the specific phases studied were of the following type: $MgCu_2$, $MgZn_2$, $MgNi_2$, $CaCu_5$ and UNi_5).

It was found that Laves phases may form in the 1·05–1·68 range of Goldschmidt radius ratios, and that the radius ratio has little influence on the structure type. The positions of the partner elements in the periodic table are the dominant factor which controls the formation of any one of the three Laves phases, but the occurrence of the phases as a group is dependent upon the ability of the partner atoms to undergo the necessary contraction or expansion to approach the effective radius ratio 1·23.

The $CaCu_5$-type compounds exist in the radius-ratio range 1·29–1·61. Below this range UNi_5-type compounds are formed.

ELECTRODEPOSITION AND OTHER COATING METHODS

Levelling Action During Nickel and Copper Plating

S. A. WATSON: 'Levelling Action During Electrodeposition in Nickel and Acid-Copper Solutions.'

Trans. Inst. Metal Finishing, 1960, vol. 37, Winter, pp. 144–55.

The work described formed an extension of the investigation of the mechanism of levelling previously reported by WATSON and EDWARDS (*ibid.*, 1957, vol. 34, pp. 167–98: abstract in *Nickel Bulletin*, 1957, vol. 30, No. 4, pp. 69–70). It was then hypothesized that levelling action is due to the greater rate of diffusion of addition agent to the peaks than to the hollows of an uneven surface, resulting in a more negative cathode potential at peaks than at hollows, and thus leading to more rapid deposition of metal in hollows than on peaks.

In support of this hypothesis the authors showed that a curve relating levelling power to concentration of levelling agent can be calculated from curves of cathode potential plotted against concentration and current density, and that the experimentally determined curve is similar to the theoretical one. The theoretical curves were, however, obtained for a nickel solution with only three addition agents. In the present paper confirmatory data are presented for other levelling agents (which were used in a nickel and an acid-copper solution), and the hypothesis is further supported by data showing the effect, on levelling, of variation in temperature and rate of agitation. The large number of addition agents studied made possible some consideration of factors which influence the effect of the additive on cathode potential during plating.

Measurements of levelling power, using microgroove record masters as substrates, were found to provide a satisfactory indication of the relative levelling action to be expected on commercially abraded surfaces. Cathode-potential measurements were made with 60 addition agents in the case of the nickel solutions, and with 30 in the case of the acid-copper solution, and where application of the hypothesis to the potential values obtained promised strong levelling action, levelling powers were measured (and proved in all such instances to be high). It is shown that a non-levelling agent, such as saccharin, can modify the behaviour of a levelling agent. Published polarographic results are empirically related to levelling action, and it is demonstrated that certain groupings in the addition-agent molecule favour levelling.

A method is described which permits rapid identification of levelling agents, by means of a simple cathode-potential change, ΔE_m.

Influence of Plating Temperature on the Properties of Bright-Nickel Deposits

R. J. CLAUSS: 'A Study of Variations in Certain Characteristics of Bright-Nickel Deposits with Variations in Bath Temperature.'

47th Ann. Tech. Proc., Amer. Electroplaters' Soc., 1960, pp. 105–9; disc., p. 240.

As recently as five years ago, bright-nickel-plating solutions were usually not operated at temperatures higher than 145°F. (62°C.). Nowadays, however, solutions are not infrequently operated at 160°F. (70°C.) or even higher temperatures. The investigation now reported was initiated, in view of these developments, to gain information on the effects of operating bright-nickel-plating solutions at temperatures higher than 175°F. (80°C.).

To assess the effectiveness of commercial bright-nickel-plating solutions at high temperatures, it was essential to evaluate the two classes of organic addition agent generally used: carrier brighteners, and secondary brighteners. Tests were first carried out using high-temperature solutions containing only a carrier brightener; the efficacy of the secondary brighteners was evaluated in relation to solutions containing a carrier brightener of organic sulphon type. The characteristics of the deposits obtained from these solutions (e.g., microstructure, appearance, ductility) were correlated with those of coatings deposited at normal plating temperatures, and, in view of reports that, like nickel deposits produced from high-temperature plating solutions, nickel-cobalt alloys deposited at plating temperatures in the range 130°–150°F. (55°–65°C.) exhibited satisfactory ductility after chromium plating, the microstructures of the bright-nickel deposits were compared with those of semi-bright and bright nickel-cobalt deposits.

The author concludes that developments in bright-nickel addition agents are near their limit with respect to maximum operating temperatures, and that the rise in operating temperatures in recent years to 165°F. (74°C.) is close to the maximum which present technology will permit.

The investigation of organic addition agents revealed that, although satisfactory bright-nickel deposits can be obtained at high temperatures, the effectiveness of the commercially available useful compounds is low compared with that at normal plating temperatures. Apart from higher rates of plating, the only outstanding property which appears unique to high-temperature plating is the increase in ductility exhibited by the nickel/chromium deposits.

Solution Transfer in Barrel Plating

P. GLAB, R. S. MODJESKA and S. P. GARY: 'Solution Transfer in Barrel Plating.'

47th Ann. Tech. Proc., Amer. Electroplaters' Soc., 1960, pp. 184–7; disc., pp. 251–2.

The study described was undertaken in an attempt to obtain quantitative data on barrel-plating variables which would throw light on solution movement, deposition rates, and potential gradients within the barrel.

Solution-transfer rates were studied by adding a soluble dye to plain water in a normal barrel-tank set-up and determining the rate of dye transfer obtained. In a similar manner, solution transfer was investigated as a function of barrel speed and type of load.

In complementary investigations, experiments were carried out, using nickel-, zinc-, cadmium-, brass- and copper-plating solutions, to gain information on the change in relevant variables during normal plating cycles.

The results of the test programme are deemed to warrant the following conclusions:

'1. Solution transfer in plating barrels is adequate, irrespective of principal variables in barrel operation.

'2. Plating-potential decrease in the load from the outside to the center is so sharp that the plating current density must be calculated on the basis of area of periphery rather than on the average over the whole load.

'3. Brighteners for barrel plating must perform over much wider current-density ranges, especially higher than those used in still tanks.

'4. In barrels, plating adds thickness in direct time relationships, unless factors producing excessive polarization are present.'

Application of Statistical Quality Control to Plating Processes

R. W. STEINMETZ: 'Applying Statistical Quality Control to Plating Processes.'

47th Ann. Tech. Proc., Amer. Electroplaters' Soc., 1960, pp. 45–50; disc., pp. 228–9.

In this paper, the author illustrates the application of statistical quality control (a scientific method of analyzing data relating to a specific process, and employing the results of the analysis in the solution of practical problems) to plating processes: in particular, to control of the thickness of the deposit and the constituents of the electrolyte. No attempt is made to give a detailed account of the mathematics involved, and the practical and economic advantages of the control chart* form the main subjects of the paper.

Of the six control charts presented and discussed, three relate to nickel-plating solutions, two to copper-plating solutions and one to a gold-plating solution.

* Data collected to ensure that the constituents of a plating solution remain within a pre-determined range, or that the thickness of the deposit meets the supplier's specification, vary from analysis to analysis or from lot to lot. When plotted on graph paper, the values form a fluctuating zig-zag pattern, and the variations can be studied. By employing the Laws of Probability, it is then possible to calculate limits for any given pattern, and, when statistical limits are added to a fluctuating pattern, the result is a 'control chart'.

Adhesion of Nickel on Substrates Contaminated with Fatty Acids

P. A. BROOK: 'Fatty Acids and Adhesion of Electrodeposited Metals.'

Nature, 1960, vol. 188, Dec. 31, p. 1183.

This communication reports an investigation which, carried out within the scope of a systematic study of the influence of monolayers of adsorbed surface-active agents on the adhesion of electrodeposits, was designed to obtain information on the effects of contamination by the fatty-acid series. The acids used were the even-numbered acids of the series $CH_3(CH_2)_nCOOH$, where $n=4-16$.

After polishing, the ends of the copper substrate were treated to obtain a monolayer of the fatty acid under study. The specimens were then plated with nickel from a conventional Watts-type solution [at a pH of 4, a temperature of 40°C., and a current density of 20 amp./sq. ft. (2·16 amp./dm.²)] until a deposit of at least 0·1 in. (2·5 mm.) was obtained. After plating the specimens were tested in a Denison tensile-testing machine.

The test data indicate that adhesion is independent of the chain-length of the fatty acid when n is greater than 8 or 10; as n decreases below this value, adhesion progressively increases. This increase is considered explicable either by the increased desorption of the shorter-chain acids, resulting in the nickel's being discharged directly onto the copper lattice, or by a decrease in the long-range forces between copper and nickel as the distance increases. As yet, it has been found impossible to distinguish between these alternatives.

Adhesion of Organic Enamels to Electrodeposited Nickel

F. X. CARLIN: 'Adhesion of Organic Enamels to Electrodeposited Nickel.'

47*th Ann. Tech. Proc., Amer. Electroplaters' Soc.*, 1960, pp. 59–62; disc., pp. 231–2.

The use of organic finishes in decorative and protective applications has increased markedly in recent years. The established protective value of electrodeposited nickel, and the likelihood of its offering an ideal undercoating for organic finishes, led to the initiation of a test programme to determine the adhesion between Watts-type nickel deposits and an oleo-resinous coating typical of the organic enamels used in the can-making industry. The results of the investigation are presented in this paper.

The oleo-resinous coating was applied to nickel undercoatings with and without a nickel-oxide surface film (i.e., surfaces representative of those some time after, and immediately after, plating). In some experiments the nickel undercoating (in each condition) was treated in various aqueous solutions of organic and inorganic compounds, to determine their effectiveness in increasing the adhesion of the enamel. Full details are given of these treatments, the nickel-plating procedure and the methods used to test the nickel/enamel bond.

The results demonstrate that a nickel deposit can be effectively employed as an undercoating for organic finishes. Although the surface of oxide-free nickel (i.e., in the condition immediately after plating) was not found conducive to satisfactory adhesion, only relatively simple treatments were necessary to modify the surface and promote adequate bonding. The simplest of these was the formation of an oxidized surface, a modification which can be accomplished merely by ensuring a sufficiently long interval between the plating and the enamelling process. Where this delay is impractical, oxide and sulphide (and possibly chromate and phosphate) films can be rapidly formed; treatments enhancing bonding characteristics are outlined.

Influence of Nickel Coatings on the Susceptibility of Cadmium-Plated Steel to Embrittlement

See abstract on p. 68.

Corrosion and Electrode-Potential Studies of Nickel/Chromium Coatings

A. H. DUROSE: 'Corrosion and Electrode-Potential Studies of Nickel-Chromium Coatings.'

47*th Ann. Tech. Proc., Amer. Electroplaters' Soc.*, 1960, pp. 83–9; disc., pp. 238–9.

Atmospheric- and accelerated-corrosion tests on nickel/chromium plated parts have indicated that corrosion-resistance can be increased by use of duplex-nickel coatings. In this paper, 'a progress report on this general subject', the author collates data available from atmospheric-corrosion tests, and attempts an explanation of the results in terms of potential, galvanic and polarization data.

The subject is considered in sections concerned with:

(1) Corrosion data on various nickel/chromium coatings (on steel or zinc substrates) incorporating duplex-nickel, bright-nickel, semi-bright-nickel or buffed-Watts-nickel deposits.

(2) Corrosion data on foil specimens of various types of nickel electrodeposit (some with a chromium overcoating).

(3) Data derived from measurements (in solutions simulating accelerated-corrosion media) of static (open-circuit) potentials for chromium and sulphur-containing and sulphur-free nickel deposits.

(4) Galvanic-couple experiments in which, to compare relative static potentials with corrosion rates, wires heavily plated with semi-bright nickel, bright nickel and chromium were immersed in three corrosive media and all three electrodes externally connected.

(5) Anodic and cathodic polarization studies of bright-nickel, semi-bright-nickel and chromium deposits.

The service and corrosion-test data are regarded as showing that duplex nickel provides better corrosion protection on steel than the same thickness of semi-bright or bright nickel. Other data (not included in the paper) indicate that the same conclusion is applicable to zinc die castings and aluminium: the use of duplex nickel has conferred excellent corrosion-resistance on aluminium. It is also demonstrated that better protection is achieved with the thicker sulphur-free nickel deposits (such as Watts or semi-bright nickel) than with the same thickness of sulphur-containing bright nickel. The relatively poor corrosion-resistance of bright nickel is thought to be attributable to its sulphur content.

The potential- and galvanic-test results show that, in most cases, bright nickel would be expected to corrode more rapidly than sulphur-free nickel or chromium. That the potential differences between chromium and the several types of nickel vary with the corrosive solution used suggests that there are variations in potential differences due to different atmospheres. This effect would be demonstrated by different degrees and types of corrosion, and is actually observed.

Corrosion-Resistance of Nickel/Chromium-Plated Zinc Die Castings

W. H. SAFRANEK, H. R. MILLER, R. W. HARDY and C. L. FAUST: 'New Data on the Performance of Nickel and Chromium Plated Zinc Die Castings.'

47*th Ann. Tech. Proc., Amer. Electroplaters' Soc.*, 1960, pp. 96–104; disc., p. 240.

Various investigations have indicated that duplex-nickel electrodeposited coatings confer greater protection against corrosion than a single layer of bright nickel, and that a thicker-than-normal chromium overcoating (deposited in crack-free form) is more effective than a conventional 0·01-mil-thick layer of chromium. Duplex-nickel coatings have been widely adopted as the nickel constituent in nickel/chromium plating zinc die-cast automobile parts, and some plants have introduced the thicker chromium finish. Composite coatings incorporating both types of deposit have been included in a three-year test programme, which was initiated (at Battelle Memorial Institute) with the aim of developing improved finishes for zinc die castings. In this paper, the authors report the progress achieved to date.

The report is in six sections. In the first, the authors discuss atmospheric- and accelerated-corrosion data tabulated on coatings comprising copper+duplex or bright nickel+LTLR* or crack-free HTHR† or duplex chromium. The second section is concerned with the endurance values and endurance indices derived for the various systems studied, while in the subsequent two sections an outline is given of the salient characteristics of duplex nickel and of thicker-than-normal crack-free chromium. The advantages of duplex-nickel/crack-free-chromium coatings, and the characteristics of bright-nickel/microcracked-chromium coatings, form the subjects of the fifth and sixth sections.

The authors regard the data presented in the paper as demonstrating the feasibility of markedly improving the endurance of zinc die castings by deposition of the following three composites (to the minimum thicknesses given):

(1) Copper 0·3 mil+duplex nickel 1·2 mil+chromium 0·01 mil.

(2) Copper 0·3 mil+duplex nickel 0·8 mil+crack-free chromium 0·03–0·05 mil.

(3) Copper 0·3 mil+bright nickel 0·8 mil+microcracked chromium 0·1–0·15 mil.

The endurance of composite (1) was 17 times greater in accelerated-corrosion studies than that of a composite consisting of 0·4 mil copper+0·8 mil bright nickel+0·01 mil chromium (which was the customary commercial plating practice during the period 1955–57). Surface pitting detrimental to appearance must, however, be expected in the case of this coating.

Data on bend-stressed panels indicate that coatings (2) and (3) exhibit superior resistance to cracking, and resistance to cracking is regarded as contributing to the durability of nickel/chromium deposits under service conditions.

Experience with the CASS Accelerated-Corrosion Test

J. D. THOMAS, D. W. HARDESTY and C. F. NIXON: 'A Progress Report on Experiences in Improving Corrosion-Resistance of Automobile Parts.'

47*th Ann. Tech. Proc., Amer. Electroplaters' Soc.*, 1960, pp. 90–5; disc., pp. 239–40.

The work reported represents a continuation of that described in the 46*th Annual Technical Proceedings of the American Electroplaters' Society* (see abstract in *Nickel Bulletin*, 1960, vol. 33, No. 4, pp. 73–4). The latter paper outlined experience with the copper-chloride-modified acetic-acid/salt-spray (CASS) test, and presented evidence in support of General Motors Corporation's decision to adopt the test as a method of evaluating the corrosion-resistance of plated automobile components. Correlation of CASS-test results with service performance has continued, and, since data available to date from the 1960 car survey suggest that the test tends to overestimate corrosion-resistance, it seemed logical to consider increasing the test time and/or acceptability standard. The present paper is concerned with the work carried out to improve the reliability and reproducibility of the test, and to gain an understanding of the corrosion processes occurring during testing.

Experiments carried out to improve control over the test environment are first described. The authors then discuss data derived from studies carried out (by analysis of the run-off solution from the surface

* Low-Temperature, Low-Ratio.

† High-Temperature, High-Ratio.

of steel test panels plated with uniform thicknesses of copper and nickel and varying thicknesses of chromium) to throw light on: (1) the corrosion mechanism involved in the CASS test, and (2) the enhanced corrosion-resistance reported to be associated with the use of chromium overcoatings greater than 0·00003 in. (0·00075 mm.) thick. From the results of these tests the authors conclude that 'there is a relationship between the amount of nickel found in the run-off solution and CASS test performance in terms of basis-metal protection afforded, and that, for chromium-plated nickel, the rate of corrosion of nickel is greatly influenced by ratio of nickel to chromium area. When this ratio is low, as appears to be the case with 'normal' chromium 0·00001 ±0·000005 inch thick, the rate of nickel penetration is great—more than a mil per 16 hours —although the total amount of nickel dissolved, and found in the run-off, is low. As the nickel-to-chromium ratio increases to infinity, the nickel penetration rate slows, for both wrought and plated Watts nickel, to one mil per 100 to 130 days, or 2400 to 3200 hours.'

In the concluding section of the paper details are given of a method developed for preparation of reproducible test samples.

Deposition and Performance of Duplex-Chromium Coatings

W. E. LOVELL, E. H. SHOTWELL and J. BOYD: 'Experience in the Operation and Performance of Dual-Chromium Systems.'

47th Ann. Tech. Proc., Amer. Electroplaters' Soc., 1960, pp. 215–25; disc., pp. 255–6.

This review of the work involved, and the success achieved, in the development (by the Ternstedt Division of General Motors Corporation) of a duplex-chromium system suitable for use as a final finish for copper/nickel-plated automobile components can be divided into four sections.

In the first the authors discuss the factors which prompted conversion to duplex chromium, and the experimental work which preceded the changeover.

The second section is concerned with data (obtained from atmospheric-corrosion and CASS tests) indicating the improved corrosion-resistance achieved by production-plating steel parts with a duplex-chromium (*vis-à-vis* a single-chromium) coating.

In the third section the salient features of the copper/nickel/duplex-chromium system, and the solutions used in its production, are considered, and emphasis is laid on the importance of achieving an appropriate micro-crack pattern in the final chromium layer.

In attributing the protection conferred by the duplex chromium to sacrificial corrosion of the underlying nickel, the authors allude to the problem entailed in the appearance, after long atmospheric exposure, of a white stain associated with nickel corrosion.

In the final section of the paper attention is drawn to the results of experiments carried out to study the corrosion mechanism of the copper/nickel/duplex-chromium system.

Corrosion Characteristics of Chromium Overcoatings

E. J. SEYB: 'Corrosion Protection with Decorative Chromium.'

47th Ann. Tech. Proc., Amer. Electroplaters' Soc., 1960, pp. 209–14; disc., p. 255.

In recent years the contribution of the chromium overcoating to the corrosion-resistance of copper/nickel/chromium decorative coatings has been the subject of much investigation. In the present paper the author's main concern is with bright crack-free-chromium and duplex-chromium coatings, which are discussed in terms of (1) atmospheric-corrosion data indicating the improved protection associated with their use as overcoatings, and (2) the mechanism underlying this improvement. Attention is, however, also drawn to the corrosion characteristics of bright-nickel/chromium, duplex-nickel, and chromium/nickel/chromium systems.

The atmospheric-corrosion results reported are regarded as substantiating the reported increase in basis-metal protection obtainable by use of thicker chromium over regular or duplex nickel.

The review of corrosion mechanisms shows that the protection conferred by bright crack-free chromium is associated with the provision of a barrier impervious to atmospheric corrosion, and is not dependent on the sacrificial corrosion of the underlying nickel; that conferred by duplex chromium, duplex nickel and buffed Watts nickel does, on the other hand, depend on sacrificial nickel corrosion.

Electroless Deposition of Nickel-Phosphorus Alloys from Pyrophosphate Solutions

M. SCHWARTZ: 'The Chemical Reduction of Nickel-Phosphorus Alloys from Pyrophosphate Solutions.'

47th Ann. Tech. Proc., Amer. Electroplaters' Soc., 1960, pp. 176–83; disc., pp. 249–50.

The alkaline electroless nickel-plating solutions suffer from two disadvantages: (1) the rate of deposition, though capable of being maintained quite constant by periodic replenishment is relatively low [of the order of 0·2–0·3 mil./hour (0·005–0·008 mm./hour)]; (2) the high temperatures employed (90°C. or above) make the solutions difficult to control, as well as inconvenient and disagreeable due to the rapid loss of ammonia. In the work outlined in the present paper, it was found that the use of pyrophosphates as complexing agents permits operating temperatures to be substantially lowered and deposition rates to be increased.

The composition of the deposits obtained from the alkaline pyrophosphate electroless nickel-plating solutions discussed is relatively constant (5 per cent. phosphorus, 95 per cent. nickel) over a wide range of solution composition and operating conditions. Data on the effects of the important solution and plating variables are presented and considered by the author, who, in summary, draws the following conclusions:

'On the basis of the work presented in this paper, the following composition and conditions are recommended for practical, commercial use, with indicated ranges:

	g./L.
Sodium pyrophosphate ($Na_4P_2O_7$)	50 (25–100)
Nickel sulphate ($NiSO_4.6H_2O$) ..	25 (10–35)
Sodium hypophosphite ($NaH_2PO_2.H_2O$)	25 (10–50)

Ammonium hydroxide (NH_4OH): to maintain pH.
Temperature: 65°–75°C.(50°–80°C.)
150°–167°F.(122°–176°F.)
pH: 10–11 (8·5–11)
Approximate deposition rate:
0·6 mil/hr. (0·2–1·0 mil/hr.)
0·015 mm./hr. (0·006–0·025 mm./hr.)

'It is suggested that the increased 'activity' of the ammoniacal pyrophosphate solution may be due to the type of complex formed and the effects of variables such as temperature and pH on the strength of this complex.'

Electrodeposition and Magnetic Properties of Cobalt-Nickel Alloys

V. ZENTNER: 'Magnetic Properties of Electrodeposited Cobalt-Nickel Alloys.'

47th Ann. Tech. Proc., Amer. Electroplaters' Soc., 1960, pp. 166–9; disc., pp. 247–9.

Electrodeposited cobalt-nickel alloys are used extensively as permanent-magnetic coatings on memory drums in digital computers. Hughes Aircraft Company has made successful use of this electrodeposited alloy in a digitair computer, which forms a vital part of the latest and most advanced fire-control system produced by the Company for the U.S. Air Force. Since saving in weight is of prime importance, the memory drum is manufactured from an aluminium alloy. After receiving a substantial copper undercoat, the component is plated with the cobalt-nickel alloy (containing about 80 per cent. cobalt).

To achieve consistent and reproducible magnetic characteristics, strict control of all the plating conditions and variables is imperative. It became clear in the early stages of the investigation of the plating variables now described that, apart from the conventional deposition variables, the type of current employed in depositing the alloy was a very important factor in relation to the magnetic characteristics of the coating. A study of the influence of superimposed a.c. current during deposition indicated that a 60-cycle a.c. current superimposed on the d.c. at a minimum ratio of 3/1 was essential to increase the coercive force, Hc, of the deposit (by about 50 per cent. compared with the straight d.c. deposit). The straight d.c. deposit has a coercive force of 200 oersteds and a remanence of 7900 gausses, while the deposit produced under the same conditions, but with a superimposed a.c. at a ratio of 3/1, exhibits a coercive force of 290 oersteds and a remanence of 4400 gausses.

The following plating conditions were found consistently to give deposits exhibiting an optimum combination of coercive force and remanence:

Solution composition

	g./L.
cobalt (as chloride)	50
nickel (as chloride)	50
boric acid	40
pH	4·9–5·1
Bath temperature	67°–72°C.
Current density	100 amp./sq. ft. (10·8 amp./dm.²)

Ratio of superimposed a.c. current to d.c. plating current: 3/1.

Deposition of Cobalt-Nickel Alloys from Sulphamate Solutions

R. C. BARRETT: 'Plating of Nickel, Cobalt, Iron and Cadmium from Sulfamate Solutions.'

47th Ann. Tech. Proc., Amer. Electroplaters' Soc., 1960, pp. 170–5; disc., p. 249.

The widening range of applications found by nickel-sulphamate plating solutions (especially where the deposit is required to exhibit low tensile stress) has prompted investigations of the feasibility of depositing metals other than nickel from acid sulphamate solutions. In this paper the author summarizes experimental data on, and solutions and conditions developed for, plating cadmium, cobalt-nickel alloys, and iron from sulphamate baths.

Plating conditions suggested for deposition of cobalt-nickel magnetic alloys are reproduced below.

Sulphamate Cobalt-Nickel-Alloy Plating Solution

	Oz./gal.	g./L.
Cobalt sulphamate	30	225
Nickel sulphamate	30	225
Boric acid	4	30
Magnesium chloride	2	15
Anti-pit agent	0·05	375 mg./L.
Co-Ni metal ratio	1:1 (approx.)	
Co-Ni alloy plating ratio	80% Co 20% Ni	
Anodes (special cast)*	80% Co 20% Ni	
pH range	2·0–4·0 (Electrometric)	
Temperature	Room, 25°C.	
Average current density	15–30 amp./sq. ft. (1·62–3·24 amp./dm.²)	
Agitation	Cathode rotation or linear movement	
Cathode efficiency	98–99%	
Anode efficiency	100%	
Appearance of deposit	Blue-white in thin films; dark matte in thicker deposits	
Stress (0·0006 in.)	14,600 p.s.i. tensile	

* Anodes can be rolled depolarized nickel, with maintenance of cobalt-nickel ratio in solution by analysis and introduction of cobalt sulphamate as required.

Metal Wear by Scoring

See abstract on p. 70.

Faraday's Laws Applied to Cleaning of Basis Metals

A. K. GRAHAM: 'Faraday's Laws Applied to Cleaning.' *47th Ann. Tech. Proc., Amer. Electroplaters' Soc.*, 1960, pp. 41–4; disc., p. 228.

In this paper, the author suggests the use of Faraday's laws as a means of developing suitable electrolytic cleaning cycles, discusses the main factors involved in such an approach, and then outlines some of the successful pre-plating treatments which have been achieved on this basis.

Six applications are discussed: (1) treatment of steel equipment prior to electrodeposition of thick nickel coatings to meet the requirements of nuclear-energy plant; (2) treatment of buffed Watts nickel coatings prior to deposition of a coating of the same type of nickel; (3) treatment of nickel-plated components (prior to re-nickel plating after stripping the chromium overcoating and polishing out the defects); (4) treatment, prior to re-nickel plating, of re-runs in which the steel substrate has become exposed through the nickel coating; (5) pre-plating treatment of soldered brass parts; (6) treatment of 'Hastelloy C', 'Stellite' and 'Carballoy' substrates prior to nickel plating. In each case, the cleaning cycles ultimately evolved are tabulated.

Cleaning Techniques in the Electronics Industry

See abstract on p. 56.

Response of Low-Alloy Steels to the A.R.D.E. Smoothing Process

See abstract on p. 69.

NON-FERROUS ALLOYS

Report of the Royal Mint: Nickel-containing Coinage

'Royal Mint: Ninetieth Annual Report of the Deputy Master and Comptroller for the Year 1959.' Published by *H.M. Stationary Office*, 1960; 87 pp.+ index. Price 5/6.

The report includes the usual statistical information on United Kingdom silver, cupro-nickel, nickel-brass and bronze coinage, currency withdrawn, assays made, and coinages produced for overseas countries. Details of the activities of the associated Mints in Australia are also given.

Coinage of completely new design struck at the Royal Mint during 1959 included a series of silver, cupro-nickel and bronze coins for Iraq, and the following series for Nigeria: cupro-nickel: 2s., 1s., 6d.; nickel-brass 3d.; bronze 1d., ½d. These coins, together with others, and with the medals and seals struck during the year, are illustrated.

The evolution of modern coinage, and the suitability of metals and other materials for use in coinage, form two of the subjects discussed by the Comptroller in his report.

Yield-Point Phenomena in Copper- and Nickel-base Alloys

R. B. JONES and V. A. PHILLIPS: 'Yield-Point Phenomena in a Number of Commercial Copper Alloys and One Nickel-base Alloy.'

Amer. Soc. Metals, 1960, *Preprint No.* 219.
Trans. Amer. Soc. Metals, 1961, vol. 53, pp. 603–20.

Previous investigations have shown that, although yield points can be produced in a variety of aluminium alloys by strain-ageing, the presence of an initial yield point in the fully recrystallized unstrained condition is comparatively uncommon. The object of the work described was 'to look at copper- and nickel-base alloys believed to show yield points, to see whether their behaviour was similar to that of aluminium alloys already studied'.

Phosphor bronze, manganic nickel, a copper-chromium alloy and a copper-beryllium alloy were studied (the first two being solid-solution alloys, and the last two age-hardenable). Particular care was taken to examine the specimens in a fine-grained condition. Data were obtained on the lower yield stress and elongation.

Initial yield points were found in the two solid-solution alloys in the fine-grained recrystallized condition, and these could be suppressed by quenching from a suitable temperature. No initial yield points were observed in the other alloys, either in the solution-treated or solution-treated-and-aged conditions. Yield points could be produced by suitable strain-ageing treatments in all the alloys.

See also

Yield-Point and Order-Hardening Phenomena in Nickel Silvers

V. A. PHILLIPS and R. B. JONES: 'Yield-Point and Order-Hardening Phenomena in Some Commercial 'Nickel Silver' Alloys.'

Amer. Soc. Metals, 1960, *Preprint No.* 231.
Trans. Amer. Soc. Metals, 1961, vol. 53, pp. 775–91.

Lüders bands are known to occur during deep drawing of the alpha copper-nickel-zinc (nickel silver) alloys. Previous work by PHILLIPS *et al.* on aluminium, copper and nickel alloys (see, *inter alia*, paper referred to in the previous abstract) demonstrated that large initial yield points in the annealed condition are relatively uncommon, unlike strain-ageing yields. The initial yield point reported to occur in nickel silver seemed worthy of investigation,

as a means of establishing whether or not the behaviour is typical of that encountered in aluminium-magnesium, copper-tin and nickel-manganese alloys. (The order-hardening phenomena reported in this paper has, it is stated, not been described previously. Ordering is not shown in the phase diagram, but the authors consider that there is now convincing evidence that ordering occurs.)

Tensile tests were carried out on three commercial grades of nickel silver: 10 per cent., 18 per cent. and 27 per cent. The authors discussion of the data recorded leads them to seven conclusions:

'1. Alpha 'nickel silver' alloys show a marked tendency to long-range order, particularly in an alloy near the Cu_2NiZn composition containing 18 per cent. nickel.

'2. Ordering results in hardening and in an increase in resistivity.

'3. The critical temperature for ordering in an 18 per cent. nickel-25 per cent. zinc-copper alloy is about 450°C.

'4. Cold working destroys ordering in 18 per cent. 'nickel silver', reducing the resistivity.

'5. The three alpha 'nickel silver' alloys tested show initial yield points with 1-25 per cent. yield elongation in the fine-grained recrystallized condition.

'6. Coarsening the grain-size decreases the magnitude of the initial yield point, but does not remove it.

'7. The grain-size-independent part of the initial yield point is attributed to ordering, the remainder to equilibrium grain-boundary segregation of solute.'

Low-Temperature Mechanical Properties of Structural Materials: Compilation of Published Data

See abstract on p. 54.

Electrodeposition and Magnetic Properties of Cobalt-Nickel Alloys

See abstract on p. 61.

Deposition of Cobalt-Nickel Alloys from Sulphamate Solutions

See abstract on p. 61.

CAST IRON

Properties of Alloy Cast Irons

R. BARTON: 'Special Cast Irons.'

B.C.I.R.A. Jnl., 1960, vol. 8, Nov., pp. 857-82.

In a previous paper the author discussed the general effect of the common alloying elements on cast iron (see abstract in *Nickel Bulletin*, 1960, vol. 33, No. 10, p. 242). Emphasis was then placed on small additions which do not greatly affect the microstructure of the iron: the influence of larger alloying additions (by which the microstructure may be changed, and greatly improved mechanical properties and enhanced resistance to heat, wear and corrosion achieved) are considered in the present review.

The characteristics of six special cast irons are discussed: high-chromium irons, and 'Ni-Hard' (which solidify according to the iron/iron-carbide system); and 'Silal', high-silicon cast irons, 'Nicrosilal', and 'Acicular Cast Iron' (which solidify according to the iron/graphite system). The high-nickel cast iron 'Ni-Resist' has been the subject of another paper by the author (see *Brit. Cast Iron Research Assocn., Jnl. Research and Development*, 1958, vol. 7, Aug., pp. 299-315: abstract in *Nickel Bulletin*, 1958, vol. 31, No. 11, pp. 292-3) and is therefore not included in the present review.

Each class of iron is considered in relation to: constitution; influence of alloying additions; physical and mechanical properties (and, where relevant, heat-, corrosion- and wear-resistance); heat-treatment; and production methods. Typical microstructures are illustrated.

Properties of Spheroidal-Graphite Alloy Cast Irons

J. GRILLIAT and R. POIROT: 'Some Spheroidal-Graphite Alloy Cast Irons, with Particular Reference to Austenitic Cast Irons.'

Fonderie, 1960, vol. 178, Nov., pp. 449-61.

Cast irons treated with magnesium to spheroidize the graphite combine the high mechanical properties associated with the spheroidal form of the graphite with the characteristics conferred by the presence of graphite *per se*. In the case of alloy cast irons, the effect of the spheroidal graphite is particularly beneficial, since it enables maximum advantage to be taken of the influence of the alloying additions on the properties of the metallic matrix.

This review of the properties and applications of the principal types of spheroidal-graphite alloy iron is in four sections. In the first the authors are concerned with the characteristics of those irons characterized by high mechanical properties or hardness [pearlitic irons for large castings; nickel-molybdenum bainitic (acicular) cast irons; normalized cast irons; quenched-and-tempered irons; air-hardening irons]. Austenitic spheroidal - graphite irons, which offer an optimum combination of castability, corrosion-resistance and mechanical properties, form the main subjects of the review, and their mechanical and corrosion-resistant characteristics, heat-treatment and applications are exemplified in some detail (in relation to the various grades of the austenitic iron 'S.G. Ni-Resist') in the second section. The suitability of spheroidal-graphite alloy irons for use at high temperatures is discussed in the third section of the paper (three classes are covered: low-alloy irons, silicon irons

and the heat-resisting grades of 'S.G. Ni-Resist'), and in the final section the authors draw attention to irons suitable for special applications, requiring, for example, non-magnetic properties or suitable coefficients of thermal expansion.

The authors' textual comments are illustrated by numerous tabular and graphical data and by photographs of typical components for which the irons are used.

Hot-Hardness of Steels and Spheroidal-Graphite Cast Iron

E. R. PETTY and H. O'NEILL: 'The Hardness of Iron, Low-Carbon Steels, and Cast-Iron Roll Materials at Various Temperatures.'

Jnl. Iron and Steel Inst., 1961, vol. 197, Feb., pp. 141–5.

The phenomenon of 'blue heat' has been the subject of much investigation ever since Stromeyer suggested that nitrogen was a cause of strain-ageing effects in commercial mild steels, and Brinell showed a peak in ball hardness values in the region of 300°C. The magnitude and position of the peak on the temperature scale is known to vary. Since pyramid hot-hardness tests with geometrically similar indentations have previously given interesting results, further work has been undertaken and is reported in this paper. Because of the shortage of data on the hot-hardness of steels and spheroidal-graphite cast irons, tests were carried out at temperatures up to 950°C. The tests were conducted on pure iron, aluminium-stabilized and rimming steels, a manganese austenitic steel, three nickel-containing spheroidal-graphite cast irons, and specimens cut from the surface of rolls produced from cast irons of the composition and structure noted below.

Total Carbon	Si	Ni	Cr	Mo	Structure
3·06	0·67	—	—	0·20	Carbide in pearlite matrix
3·16	1·17	3·68	0·12	0·75	Acicular S.G. rolls
3·01	1·10	3·68	0·09	0·92	Acicular S.G. rolls
2·16	0·52	0·69	1·05	—	Carbide in a spheroidized pearlite matrix
2·11	0·55	0·51	13·0	—	Experimental roll

'Blue hardening' is discussed in relation to the hot hardness data obtained, and to tensile and other results available in the literature. It is concluded that the hardness peak at 250°C. is a reflexion of an increase in strain-hardening capacity. That a 'blue-hardening' peak also obtains for spheroidal-graphite cast irons, but not for austenitic manganese steel, is deemed of interest. The spheroidal-graphite cast irons conform, at least at elevated temperatures, to the known relationship between hardness and ultimate tensile strength for steels.

The hot-hardness tests on the cast-iron roll materials explain why it is difficult, if not impossible, to relate the life of rolls to their room-temperature hardness. The rolls soften to different degrees at temperatures in the working range.

Low-Temperature Mechanical Properties of Structural Materials: Compilation of Published Data

See abstract on p. 54.

Low-Temperature Properties of Ferrous Materials

See abstract on p. 54.

Metal Wear by Scoring

See abstract on p. 70.

Combined Method for Analysis of High-Alloy Irons

See abstract on p. 80.

CONSTRUCTIONAL STEELS

Vacuum-Induction Melting of High-Strength Steels

P. S. SCHAFFER, P. J. AHEARN and M. C. FLEMINGS: 'Vacuum-Induction Melting High-Strength Steels.'

Modern Castings, 1960, vol. 38, Oct., pp. 95–112.

The production of superior-quality high-strength steels by vacuum-melting techniques requires an understanding (1) of the thermodynamics and kinetics involved in steel-making reactions, and (2) of the effect of reduced pressure on these reactions. The present paper comprises 'a detailed account of the theoretical aspects, experimental procedures and results obtained by vacuum- and controlled-atmosphere melting, refining and casting of high-strength steels, to yield low residual-gas content and improved cleanliness'.

The paper is in two sections. In the first, the authors discuss the theoretical aspects of crucible/melt reactions, deoxidation, desulphurization, degassification, dissociation and volatilization. In the second, they outline the procedures used, and the results obtained, in applying the findings of the theoretical review to vacuum-induction melting and casting A.I.S.I. 4340 nickel-chromium-molybdenum high-strength steel.

Vacuum-induction melting was found appreciably to reduce the residual gas content and the size and frequency of non-metallic inclusions, an effect which resulted (compared with steel castings produced by conventional air-melting methods) in higher ductility and toughness, especially at ultra-high-strength levels.

Size Effects in Notched-Bend Tests on Low-Alloy Steels

S. YUKAWA: 'Temperature and Microstructure Dependence of Size Effects in Notched-Bend Tests of Some Alloy Steels.'

Amer. Soc. Metals, 1960, *Preprint No.* 238.
Trans. Amer. Soc. Metals, 1961, vol. 53, pp. 871–81.

Work by LUBAHN and YUKAWA has demonstrated the importance of notch acuity in relation to the decrease in strength of a nickel-molybdenum-vanadium heat-treated steel as a result of increase in size of notched-bend specimens (see *Proc. Amer. Soc. Testing Materials*, 1958, vol. 58, p. 661). Since the available data were not completely definitive in indicating the relationship between the size effect and mechanical properties, the investigation now reported was undertaken to explore the relationship in steels typical of those used in large rotor forgings ('high-hardenability steels containing 0·2–0·3 pct. C and Ni, Mn, Cr, Mo and V in various amounts as the alloying elements').

The specimens employed were square in cross-section, with a notch (45° included angle, 20 per cent. depth) across one surface at mid-length. Notch radii and specimen depth were varied. The specimens were loaded as a simple beam, with the notch in tension, at the slow rates characteristic of hydraulic testing machines. In general, the steels tested were subjected to the conventional heat-treating practices (to obtain a microstructure of tempered bainite, accompanied, in certain cases, by some pro-eutectoid ferrite), but, to determine the effect of microstructure, tests were carried out also on steel austenitized, oil-quenched and tempered. In other experiments account was taken of the effects of the test temperature.

Increase in the specimen size was found to increase propensity to sudden brittle fracture and to decrease the nominal strength (the first effect occurring at higher temperatures than the second). The results suggest that the 50 per cent. fibrous transition temperature (as measured by Charpy V-notch tests) offers a useful indication of sensitivity to the size effect. The effects of specimen size became less as the transition temperature fell, and the influence of microstructure can be inferred from its effect on transition temperature.

The results are discussed in terms of the limiting notched bend strength and the modified Griffith theory of fracturing.

Influence of Cold Work on the Structure and High-Temperature Strength of Steel

P. SHAHINIAN, M. R. ACHTER and W. A. PENNINGTON: 'The Effect of Cold Work and Temperature on Strength and Structure of Steel.'

Amer. Soc. Metals, 1960, *Preprint No.* 234.
Trans. Amer. Soc. Metals, 1961, vol. 53, pp. 817–36.

The influence of cold work on the strength of A.I.S.I. 4140 low-alloy steel at elevated temperature was investigated by hardness measurements and determination of tensile and creep-rupture properties. Its effect on microstructure was studied by optical and electron microscopy and by X-ray-diffraction techniques.

Large amounts of cold work were found to strengthen the quenched-and-tempered steel in creep-rupture at intermediate temperatures, but the steel was weakened by small degrees of deformation. The reduction in strength of the worked material is attributed to an observed increase in mean free ferrite path, apparently induced by the prior cold work. The weakening at small deformations and strengthening at large deformations are explained in terms of two opposing effects of cold work.

Low-Temperature Flow and Fracture Characteristics of Low-Alloy Steel

F. R. LARSON and J. NUNES: 'Low-Temperature Flow and Fracture Tension Properties of Heat-Treated SAE 4340 Steel.'

Amer. Soc. Metals, 1960, *Preprint* 223.
Trans. Amer. Soc. Metals, 1961, vol. 53, pp. 663–82.

The investigation reported was undertaken to provide information on the plastic-flow properties (particularly at low temperatures) of heat-treated low-alloy steel.

Two heats of A.I.S.I. 4340 low-alloy steel were selected for study:

C %	Mn %	Si %	S %	P %	Ni %	Cr %	Mo %	V %
0·370 to 0·385	0·75 to 0·77	0·27 to 0·28	0·013 to 0·015	0·013 to 0·016	1·73 to 1·77	0·83 to 0·86	0·25 to 0·27	—
0·375 to 0·385	0·75 to 0·77	0·32 to 0·33	0·020 to 0·023	0·008 to 0·009	1·75 to 1·80	0·79 to 0·80	0·24 to 0·26	Trace

Preliminary experiments on martensitic specimens at three strength levels led to a more extensive investigation of specimens subjected to heat-treatments designed to produce microstructures (tempered martensite, bainite or pearlite) varying in hardness. True-stress/true-strain tension properties were determined, at a testing speed of 0·01 in. (0·25 mm.)/minute, at temperatures ranging from +200° to −196°C. Data were obtained on strain hardening, the flow stress, fracture stress and fracture strain.

On the basis of the data presented the authors draw the following conclusions:

'1. An empirical equation can be developed which adequately describes the influence of testing temperature upon the flow stress, and can be expressed as:

$$\sigma_{0\cdot04} = M/T + \sigma_0$$

'2. The slope (M) of reciprocal-temperature *versus* flow-stress plots was nearly identical for all heat-treatments.

'3. Values for σ_0 were obtained, and were found to be some function of heat-treatment.

'4. When the data were corrected for triaxial stresses in the neck, the often-proposed equation of

$$\sigma = S_1 \varepsilon^n$$

fits the data well.

'5. The martensitic and pearlitic structures showed yield points whose magnitude was dependent on testing temperature and strength level.

'6. The bainitic structures did not show yield points at any testing temperature.

'7. Room-temperature strain hardening (or true strain at maximum load) decreases to a minimum value (about 0·04) with increasing strength level.

'8. Varying the testing temperature did not change the strain-hardening properties appreciably.

'9. The fracture strain decreases with increasing strength level and decreasing testing temperature. No precise function could be found to describe this behaviour, as it is influenced by differences in fracturing mechanism. The assumption is based upon the varied surface features of the fractured specimens studied.'

Low-Temperature Tests on Pressure-Vessels Fabricated from 9 per cent. Nickel Steel in Various Conditions of Heat-Treatment

'Why Stress Relieve Cryogenic Vessels Made from 9% Nickel Steels ?'

Welding Design and Fabrication, 1960, vol. 33, Dec., pp. 57-9.

Nine per cent. nickel steel was developed by The International Nickel Company, Inc., almost 15 years ago. At present the material is covered by *A.S.T.M. Specifications A*300 [which requires a keyhole-notch impact value of 15 ft./lb. at −320°F. (−196°C.)] and *A*353-58. The steel, produced to the requirements of these specifications, may be used for vessels constructed under the A.S.M.E. Boiler and Pressure Vessel Code for service at temperatures down to −320°F. (−196°C.), *provided that the vessels are stress relieved after fabrication.* When the steel was first developed, commercial quenching facilities for plates were limited, and emphasis was placed on the production of good low-temperature properties by normalizing. The treatment specified under *A*353 involves two normalizing treatments: the first from 1650°F. (900°C.), and the second from 1450°F. (785°C.). Development of optimum low-temperature properties necessitates a third treatment [tempering at 1025°-1085°F. (550°-585°C.)]. With the introduction of continuous heat-treating facilities for the production of quenched-and-tempered plates under uniform and closely controlled conditions, it was decided, since quenching and tempering is a considerably simpler heat-treatment than double normalizing and tempering, to investigate the properties of 9 per cent. nickel steel in the quenched-and-tempered condition. The results of the tests subsequently carried out on pressure vessels filled with liquid nitrogen are discussed in this article.

Operation Cryogenics (as the test programme was named) was conducted jointly by The International Nickel Company, Inc., U.S. Steel Corporation and Chicago Bridge and Iron Company. Seven pressure vessels were tested to destruction. Two were of rectangular design, simulating a type used for ship-board transport of liquid gases. One of these was fabricated from steel water-quenched from 1475°F. (800°C.) and tempered at 1100°F. (595°C.); the other was fabricated from double-normalized and tempered material. Both vessels were tested as-welded. The vessels were filled with liquid nitrogen at −320°F. (−196°C.), pressurized to 80 p.s.i., and subjected to the repeated impact of a steel-wrecking ball, which was swung from heights of up to 19 ft. (6 m.) against the side of the vessel [to deliver blows in excess of 80,000 ft. lb. (11040 kgm.)]. In each case failure occurred as a crack at one of the vertical corners near the top stiffeners. After the crack had developed, each vessel was struck at least once, but the crack was not found to propagate.

In the second phase of the programme five cylindrical vessels (representing a type used for land-based storage of liquid gases) were filled with liquid nitrogen at −320°F. (−196°C.), and the pressure was increased until they failed by bursting. Two were fabricated from quenched-and-tempered steel, the others from double-normalized and tempered material. One of the former type, and two of the latter, were stress-relieved in accordance with the A.S.M.E. Boiler and Pressure Vessel Code. The burst stresses of all the vessels were found to be at least four times the design stress permitted.

The results of the test programme demonstrate that 9 per cent. nickel steel exhibits, in the quenched-and-tempered condition, low-temperature notch toughness equal or superior to that of the double-normalized and tempered material called for in present Code requirements. The participating companies consider that acceptance of the steel in the quenched-and-tempered condition would make it less costly to produce, consequently less expensive to the user, and hence more attractive to designers and fabricators.

The steel is notch-tough with either heat-treatment, and will resist brittle fracture even in the presence of the pronounced residual stresses found in as-welded vessels. This finding should, it is felt, make it possible to omit the stress-relief treatment now required after welding.

See also

''Operation Cryogenics' Demonstrates Toughness of 9% Nickel Steel.'

INCO Nickel Topics, 1961, vol. 14, No. 1, pp. 2-3.

This article, which, like that referred to in the preceding abstract, reviews the results of Operation Cryogenics, includes a table summarizing data derived from cylindrical pressure vessels subjected

Results of Bursting Tests on 9 Per Cent. Nickel Steel Pressure Vessels

(*See abstract on p. 66*)

Test No.	PC-1	PC-2	PC-3	PC-4	C-5	PC-6
Treatment	Q. and T., As-Welded	Double Norm., Tempered, Stress-Relieved	Q. and T., Stress-Relieved	Double Norm., Tempered, As-Welded	Q. and T., As-Welded	Double Norm., Tempered, Stress-Relieved
Temp. of vessel at start of test	−308°F. −188°C.	−320°F. −196°C.	−320°F. −196°C.	−320°F. −196°C.	−320°F. −196°C.	−320°F. −196°C.
Temp. at failure	−304°F. −186°C.	−320°F. −196°C.	−311°F. −191°C.	−310°F. −190°C.	−302°F. −185°C.	−314°F. −192°C.
Burst pressure	2275 p.s.i. (159 kg./cm.²)	1550 p.s.i. (109 kg./cm.²)	2125 p.s.i. (149 kg./cm.²)	2300 p.s.i. (161 kg./cm.²)	2160 p.s.i. (151 kg./cm.²)	2110 p.s.i. (148 kg./cm.²)
Increase in diameter*	0·29%	0·21%	0·29%	0·40%	—	—
Burst Stress	135,510 p.s.i. (9486 kg./cm.²)	92,920 p.s.i. (6504 kg./cm.²)	129,800 p.s.i. (9086 kg./cm.²)	135,730 p.s.i. (9501 kg./cm.²)	132,000 p.s.i. (9240 kg./cm.²)	128,000 p.s.i. (8960 kg./cm.²)

* Original diameter = 47·99 in. (120 cm.); thickness = 0·405 in. (1·02 cm.).

to bursting tests during Operation Cryogenics and a previous investigation. This table is reproduced above.

See also

'Welded Nickel-Steel Vessels Withstand Pressure, Impact Without Stress Relief.'

Welding Engineer, 1960, vol. 45, Dec., pp. 38–40.

Low-Temperature Mechanical Properties of Structural Materials: Compilation of Published Data

See abstract on p. 54.

Low-Temperature Properties of Ferrous Materials

See abstract on p. 54.

Influence of Rapid Tempering on Susceptibility to 500°F. Embrittlement

A. NAKASHIMA and J. F. LIBSCH: 'Effect of Induction on 500° Embrittlement.'

Amer. Soc. Metals, 1960, *Preprint* 229.
Trans. Amer. Soc. Metals, 1961, vol. 53, pp. 753–64.

500°F. (260°C.) embrittlement is of considerable practical interest in relation to the use of high-strength steels, and many attempts have been made to prevent its occurrence or minimize its effects. In a study of rapid tempering using induction heating (*Metal Progress*, 1950, Aug., p. 176), LIBSCH and POWERS noted some improvement in the room-temperature impact-resistance of an A.I.S.I. 3140 steel after tempering to a hardness level associated with 500°F. embrittlement. The investigation now described was conducted to obtain more information on the influence of rapid heating upon susceptibility to 500°F. embrittlement, in the hope of (1) finding some means of minimizing loss of notch-toughness, and (2) contributing to an understanding of the mechanism of embrittlement.

A.I.S.I. 3140 steel,* which was chosen for study, exhibits a pronounced loss in room-temperature impact-resistance when subjected to furnace tempering in the range 500°–600°F. (260°–315°C.) for 1 to 2 hours after hardening.

All the specimens were hardened by being heated, in a protective atmosphere, to 1600°F. (870°C.) and quenched in agitated oil. After quenching they were cooled to −110°F. (−79°C.) to obtain a 100 per cent. martensitic structure. One group of specimens was then tempered in the furnace at temperatures varying from 212° to 1320° F. (100° to 715°C.) for 1 hour, while a second group was tempered by induction heating in the same temperature range 'for O seconds' at temperature. After heat-treatment, a 'V' notch was ground in the specimens and Charpy impact tests were conducted at temperatures ranging from room temperature to −310°F. (−190°C.). Transition temperatures were determined, and hardness measurements carried out. Selected specimens were subjected to X-ray-diffraction and electron-micrographic study, in an effort to throw light on the structural changes associated with tempering.

It is considered apparent from the data presented that the use of the rapid-heating and short-tempering cycles characteristic of induction tempering avoids the severe loss in notch toughness associated with furnace tempering at 500°–600°F. for 1 hour. There is, nevertheless, some tendency towards embrittlement upon induction tempering at hardness levels associated with 500°F. embrittlement. Whilst embrittlement is time- as well as temperature-dependent,

* Composition: carbon 0·43, manganese 0·85, silicon 0·29, sulphur 0·011, phosphorus 0·01, nickel 1·23, chromium 0·62, molybdenum 0·03, copper 0·1. per cent.

the data indicate that the embrittling effect may be largely suppressed if the time in the embrittling range is very short (i.e., of the order of seconds). In tempering treatments producing a hardness of less than Rc55 in A.I.S.I. 3140 steel, the transition temperature of induction-tempered specimens thus remained consistently below that of furnace-tempered specimens.

Induction tempering does not apparently fully condition the steel against embrittlement upon further tempering in the embrittlement range (furnace tempering at 485°F. (250°C.) after induction tempering caused an increase in the transition temperature).

The data suggest that 500°F. embrittlement is related to the form and distribution of the cementite precipitate upon initiation of the third stage of tempering. It is considered possible that during induction tempering high heating rates (associated with stress gradients) influence the nucleation and growth of the cementite which forms during induction tempering. A greater number of nucleation sites may contribute to a wider distribution of the cementite, while the very short heating times may minimize the growth of elongated films: embrittlement apparently proceeds under conditions conducive to growth (i.e., furnace tempering following induction tempering). It is deemed probable that very short tempering cycles also influence the formation and solution of ε-carbide which, in turn, may influence the distribution and form of the cementite produced at the initiation of the third stage of tempering.

Influence of Surface Coatings on the Susceptibility of High-Strength Steels to Stress Corrosion

See abstract on p. 75.

Symposium on Hydrogen Embrittlement

47th Ann. Tech. Proc., Amer. Electroplaters' Soc., 1960, pp. 110–65.

Educational Sessions E and F of the Forty-Seventh Annual Convention of the American Electroplaters' Society, held at Los Angeles in July 1960, consisted of a Symposium on Hydrogen Embrittlement. Considerations of space preclude inclusion of papers of no specific relationship to nickel plating or nickel-containing steels: reference to these papers (which are nevertheless of indirect interest) are listed below. The scope of the other papers is indicated in the abstracts following this list.

H. J. READ: 'The Metallurgical Aspects of Hydrogen Embrittlement in Metal Finishing', pp. 110–15; disc., 241–2.

R. J. BARTON: 'On the Mechanism of Transport of Hydrogen Across a Solution - Metal Interface,' pp. 116–23; disc., p. 242.

M. L. HILL: 'The Behaviour of Hydrogen in Iron and Steel', pp. 124–34; disc., pp. 242–3.

S. C. LAWRENCE: 'Detection and Control of Chemisorbed Hydrogen During Electrolysis', pp. 135–7; disc., p. 243.

C. A. SNAVELY: 'Measurements Pertaining to Hydrogen Embrittlement', pp. 138–42; disc., p. 244.

Determination of Hydrogen-Embrittlement Effects in Cadmium-Plated High-Strength Steels.

N. M. GEYER, G. W. LAWLESS and B. COHEN: 'A New Look at the Hydrogen Embrittlement of Cadmium-Coated High-Strength Steels', pp. 143–51; disc., pp. 244–6.

The work reported was initiated with two aims in mind: (1) to establish a test method for studying hydrogen-embrittlement effects, particularly in relation to electroplating processes; (2) to investigate various cadmium-plating processes in the light of their capacity to plate ultra-high-strength steel without inducing hydrogen embrittlement.

On the basis of the results obtained in phase (1) of the investigation, it is concluded that a sustained-load test on notched tensile specimens (in which the specimen is loaded at 75 per cent. of the ultimate notch tensile strength for a minimum of 200 hours) offers a reliable and sensitive means of determining the susceptibility of cadmium-plated high-strength steel to hydrogen embrittlement.

In phase (2), the test was used to assess the embrittling tendencies of conventional-cyanide, high-efficiency-cyanide, sulphamate, and fluoborate cadmium-plating solutions. Immersion-plating and vacuum-metallizing processes were also studied. A.I.S.I. 4340 nickel-chromium-molybdenum steel and 'Thermold J' tool steel were used as basis metals.

The data obtained show that hydrogen embrittlement is related to the basis metal, the current efficiency of the cadmium-plating solution, and the physical structure of the coating.

Influence of Nickel Coatings on the Susceptibility of Cadmium-Plated Steel to Hydrogen Embrittlement.

W. BECK and E. J. JANKOWSKY: 'The Effectiveness of Metallic Undercoats in Minimizing Plating Embrittlement of Ultra-High-Strength Steel', pp. 152–9; disc., p. 246.

The aim of the investigation described by the authors was fourfold: (1) to investigate the effectiveness of Watts-type bright-nickel and pyrophosphate-copper undercoatings in minimizing the embrittlement induced by overplating with cadmium coatings from cyanide solutions or with conventional chromium coatings; (2) to investigate the effect, on susceptibility to embrittlement, of replacing the cyanide-cadmium by a fluoborate-cadmium solution; (3) to evaluate embrittlement by various methods known to be sensitive to changes in ductility; (4) to consider the electrochemical variables responsible for the progressive increase in embrittlement induced by plating

from fluoborate-cadmium, cyanide-cadmium and chromium solutions.

A.I.S.I. 4340 nickel-chromium-molybdenum low-alloy steel was selected for study as the basis metal. Embrittlement was evaluated by determination of bend ductility and tensile ductility and by sustained-load delayed-failure tests on notched specimens.

From the results given in the paper the authors conclude that the sustained-load test is more sensitive and reliable than the other two tests studied.

Hydrogen embrittlement induced by chromium plating and cyanide-cadmium plating was substantially reduced by deposition of undercoatings of pyrophosphate copper and Watts-type nickel. Replacement of the cyanide cadmium-plating solution by the fluoborate solution also reduced embrittlement. Mechanisms are suggested in explanation of the decrease in hydrogen embrittlement associated with the use of metallic undercoatings and the fluoborate solution.

Non-Embrittling Nitrate-Containing Cadmium-Plating Solution.

W. F. HAMILTON and M. LEVINE: 'Control of Hydrogen Embrittlement by Plating from Cadmium Cyanide Baths Containing Nitrate', pp. 160–65; disc., pp. 246–7.

The authors give details of the research which culminated in the development of a modification of the conventional cadmium-plating process suitable (as indicated by data presented on A.I.S.I. 4340 nickel-chromium-molybdenum steel) for use in plating high-strength steels.

Addition of sodium nitrate and an appropriate throwing-power additive to a high-cadmium low-free-cyanide electrolyte was found to result in satisfactory operating characteristics, without visible cathodic gassing, and to obviate the need for post-plating baking.

Particulars are given of a new cleaning procedure developed for use with the modified electrolyte, and the operation and control of the process are discussed.

Metal Wear by Scoring

See abstract on p. 70.

Response of Low-Alloy Steels to the A.R.D.E. Smoothing Process

W. A. MARSHALL, V. J. SANDERS and R. A. F. HAMMOND: *Trans. Inst. Metal Finishing*, 1960, vol. 37, Winter, pp. 132–43.

In the A.R.D.E.* smoothing process the steel under treatment is exposed to an aqueous solution of oxalic acid, hydrogen peroxide and sulphuric acid, which exerts a pronounced smoothing action (see *Jnl. Electroplaters' Tech. Soc.*, 1952, vol. 28, pp. 27–46). A suitable solution contains (g./100 ml.): oxalic acid (crystals) 2·5, hydrogen peroxide 1·3, sulphuric acid 0·01. The behaviour of ferrous materials when treated by the process is known to depend largely on the nature and condition of the material. While experience in the many applications of the process has naturally afforded a good deal of useful empirical information, a systematic study of the effects of the treatment (as a function of the composition and metallurgical condition of the steel) has not hitherto been undertaken. The present paper reports the results of a series of experiments carried out to determine the response, to the smoothing solution, of a range of low-carbon, medium-carbon and low-alloy molybdenum, chromium-molybdenum and nickel-chromium-molybdenum steels in various conditions of heat-treatment.

The factors investigated included: (1) development of reflectivity, (2) changes in surface roughness, (3) rate of dissolution, (4) frequency, form and range of periodic potential pulse, (5) formation of smut. On the basis of the data presented, the authors propose tentative theories relating the behaviour of the steels to their composition and metallurgical condition.

* Armament Research and Development Establishment.

Chemical Milling

See abstract on p. 55.

HEAT- AND CORROSION-RESISTING MATERIALS

Determination of Proof Stress, Rupture Strength and Creep Strength at Elevated Temperatures: British Standards

The British Standards to which attention is drawn below form three of a series specifying requirements for obtaining the data necessary to determine the properties of steel at elevated temperatures. Each contains details of the method of testing, the selection and number of samples, the test conditions, and the presentation of results.

BRIT. STANDARDS INSTN.: 'Procedures for Obtaining Properties of Steel at Elevated Temperatures. Part 1. Proof Stress.'

B.S. 3228: *Part* 1: 1960; 7 pp. Price 3/-.

The Standard 'specifies the procedure for obtaining the range of values for any selected proof stress between 0·1 per cent. and 0·5 per cent. for a selected steel at elevated temperatures'.

BRIT. STANDARDS INSTN.: 'Procedures for Obtaining Properties of Steel at Elevated Temperatures. Part 2. Rupture Strength.'

B.S. 3228: *Part* 2: 1960; 7 pp. Price 3/-.

The Standard 'specifies the procedure for obtaining

the range of rupture strength at elevated temperatures for a selected steel in lengths of time up to 100,000 hours'.

BRIT. STANDARDS INSTN.: 'Procedures for Obtaining Properties of Steel at Elevated Temperatures. Part 3. Creep Strength.'

B.S. 3228: *Part* 3: 1960; 7 pp. Price 3/-.

The Standard 'specifies the procedure for obtaining the range of creep strength at elevated temperatures for a selected steel in times up to 100,000 hours'.

Development of Improved Nickel-base High-Temperature Alloys

J. C. FRECHE, W. J. WATERS and T. J. RILEY: 'A New Series of Nickel-base Alloys for Advanced-Temperature Applications.'

Amer. Soc. Metals, 1960, *Preprint No.* 214.
Trans. Amer. Soc. Metals, 1961, vol. 53, pp. 523–37.

The research described, initiated at the Lewis Research Center of the National Aeronautics and Space Administration in an attempt to develop a nickel-base turbine-blading alloy capable of operating at higher temperatures than those currently available, has been previously reported in greater detail elsewhere.

In the initial phase of the investigation, modification of a basis composition (molybdenum 8, chromium 6, aluminium 6, zirconium 1, wt./ per cent., remainder nickel) with small additions of boron, carbon, titanium, or titanium + carbon culminated in the development of a series of alloys exhibiting good stress-rupture properties, oxidation-resistance and impact characteristics: see *National Aeronautics and Space Administration Memorandum* 4-13-59*E* (abstract in *Nickel Bulletin*, 1959, vol. 32, No. 8, pp. 263–4).

The second phase of the test programme (in which attempts were made, using the same basis composition, to achieve greater strength at elevated temperatures by means of alloying additions of vanadium) was reported in *National Aeronautics and Space Administration Technical Note D*-260: see abstract in *Nickel Bulletin*, 1960, vol. 33, No. 8–9, p. 209.

Fatigue Data on Precipitation-Hardenable Stainless Steels

R. J. FAVOR, H. J. GROVER and W. P. ACHBACH: 'Fatigue Data on Precipitation-Hardenable Stainless Steels.'

Defense Metals Information Center, Battelle Memorial Inst., Memorandum 46, Mar. 11, 1960; 15 pp.

The fatigue data presented were culled from the literature and relate only to the semi-austenitic precipitation-hardenable steels '17-7 P.H.', 'AM-350' and 'AM-355'. The reader is reminded that 'the objectives of each memorandum are to make reported fatigue data available as quickly as these can be collected and tabulated. Towards this end, only limited time has been spent either in interpretation or in attempts to make the compilations complete.'

On the basis of the limited data presented, the authors tentatively conclude that the materials exhibit reasonably good fatigue properties in comparison with many other metals and alloys.

Properties of Alloy Cast Irons

See abstract on p. 63.

Metal Wear by Scoring

J. H. OLSON and R. D. CHAPMAN: 'Metal Wear by Scoring.'

Amer. Soc. Metals, 1960, *Preprint No.* 204.
Trans. Amer. Soc. Metals, 1961, vol. 53, pp. 359–79.

Wear by cutting, abrasion, pitting or corrosion can usually be controlled by ensuring that surfaces are smooth and free from abrasive or corrosive agents. Wear by scoring is often a more difficult problem, and even well-lubricated surfaces require score-resistance to prevent damage or possible seizure during starting and stopping or periods of overload. In the investigation described, various ferrous/ferrous and ferrous/non-ferrous combinations of flat wear specimens (lubricated and in reciprocating motion) were progressively loaded until scoring occurred.

The following types of material were selected for study: 1 per cent. chromium steel (A.I.S.I. 51100); piston-ring, cylinder-block, malleable, spheroidal-graphite, chromium-vanadium, chromium-titanium, chromium-nickel-molybdenum and chromium-molybdenum-boron cast irons; 'Armco' iron; carbon steel (A.I.S.I. 1017); 14 per cent. manganese steel; molybdenum steel (A.I.S.I. 4028); nickel-chromium-molybdenum steels (A.I.S.I. 8620 and 94B17); 18-8 austenitic steel (Type 302). The efficacy of various surface treatments (e.g., carburizing, chemical-deposition of nickel-phosphorus coatings, anodizing, sprayed coatings) in reducing scoring was also evaluated. Details are given of the apparatus developed to study scoring on flat lubricated surfaces in reciprocating motion, and the data obtained by its use in the present investigation are fully discussed.

In addition to the compatibility of the two metals, the rate of increase in temperature during testing was found to be a critical factor in determining the scoring point. This rise in temperature is, however, influenced by surface finish and thermal conductivity, and can therefore be controlled: the results show that smooth surfaces do not heat rapidly and can support heavy loads, and that good thermal conductivity, by enhancing 'primary heat dissipation', significantly reduces scoring tendencies, even though considerable heat is generated at the wear surfaces. These factors, and the rôle of surface hardness and work hardening, are discussed. Reasons are suggested for the anomalous behaviour of cast iron, which, unlike the other metals tested, scored more easily when the finish was improved.

Higher score loads can be attained, and score damage minimized, by utilizing an aluminium (rather than a steel) base under hard-plated surfaces, to enhance thermal conductivity and prevent loss of the oil film.

Factors Influencing Corrosion in Furnace Atmospheres

H. L. KEE: 'For Service Under Fire—Control Corrosion.'

Product Engineering, 1960, vol. 31, Nov. 28, pp. 44–5.

In furnaces operating at combustion temperatures higher than 1500°F. (815°C.), contaminants in the fuel, or even the fuel itself, can give rise to corrosion in the materials of construction used. In this short article (which is intended essentially as a practical guide to the problems involved), the author discusses three ways of minimizing corrosion, and hence of extending the life, of furnace components: (1) by lowering the combustion temperature, or in some way (e.g., by shielding) lowering the temperature of the component concerned; (2) by reducing the corrosiveness of the fuel (e.g., by suitable adjustment of the combustion conditions, by use of a different fuel, or by addition of inhibitors); and (3) by selecting a more corrosion-resistant material.

In the section on material selection, the author indicates (within the limits of the article's scope) the suitability of the various types of chromium-nickel heat-resisting steel and nickel-base high-temperature alloy for service in furnace atmospheres.

Anodic Passivity in Metals

K. SCHWABE: 'The Causes of Anodic Passivity in Metals.'

Electrochimica Acta, 1960, vol. 3, Oct., pp. 186–94.

On the basis of the premise that the presence of porous films does not constitute a necessary requirement for the passivation of metals (in particular, nickel and iron), the author develops a kinetic treatment of the passivation process in ferrous metals, according to which the hindered dissolution of these metals at high current densities leads to the formation of chemisorbed oxygen layers. The extent of the resulting passivation depends on the nature of the anions present in the solution, since these anions will tend to displace the oxygen with variable rapidity. The different passivation mechanisms are reviewed.

Cathodic Protection of Metals

M. POURBAIX: 'Conditions for the Cathodic Protection of Metals.'

Werkstoffe u. Korrosion, 1960, vol. 11, Dec., pp. 761–6.

After discussing the place of cathodic protection in the general field of corrosion prevention, the author discusses the thermodynamic and kinetic conditions prerequisite for the process's application. In this connexion, reference is made to diagrams indicating the values of electrode potential and pH associated with corrosion, passivity or immunity, and the significance of these diagrams in relation to cathodic protection is discussed.

The application of polarization curves to the development of cathodic-protection systems for carbon and stainless steels is exemplified, and emphasis is placed on the dangers inherent in a badly planned system. Finally the author considers the effectiveness, reliability and commercial potentiality of this type of protection.

Thermodynamics of Corrosion in Fused Chlorides

C. EDELEANU and R. LITTLEWOOD: 'Thermodynamics of Corrosion in Fused Chlorides.'

Electrochimica Acta, 1960, vol. 3, Oct., pp. 195–207.

Industrial interest in fused halides has increased in recent years, partly because of the great demand for those metals produced *viâ* their fused salts, and partly because advances in high-temperature chemical engineering and in vacuum technology have now rendered commercially feasible a number of new processes using fused salts. In this paper the authors consider, with particular reference to the corrosion of metals, the thermodynamics of the metal/molten-chloride system. It is suggested that, at the high temperatures now being considered, effective passivity will be unlikely to occur in essentially chloride melts, so that the only way of achieving the required high standard of corrosion-resistance is by ensuring that the system is near thermodynamic equilibrium. The argument is illustrated by thermal data on, *inter alia*, nickel.

It is shown that the prime factors which determine the degree to which corrosion can occur in a given system are the nobility of the metal and the stability of the chloride. Also important are the initial redox potential of the melt, the solubility in the melt of the metal produced by the reduction of the chloride, the pressure over the system, the solubility product of the metal oxide, and the presence of impurities (including water, which is considered in some detail). The authors take no account of kinetics, since the main interest is in determining the conditions under which the reaction can be expected to come to a virtual halt, and it is assumed that, at the temperatures under consideration, the rates of reactions (other than those involving extremely small concentrations) will be much in excess of those tolerable in critical applications.

Intergranular Oxidation of 18-8 Stainless Steels by Oxygen and Dry Sodium Chloride

H. W. PICKERING, F. H. BECK and M. G. FONTANA: 'Rapid Intergranular Oxidation of 18-8 Stainless Steels by Oxygen and Dry Sodium Chloride at Elevated Temperatures.'

Amer. Soc. Metals, 1960, *Preprint No.* 232.
Trans. Amer. Soc. Metals, 1961, vol. 53, pp. 793–803.

The mass of literature published on the susceptibility of austenitic stainless steel to stress-corrosion

cracking is concerned primarily with behaviour in aqueous environments. A similar type of corrosion has, however, been reported to occur in titanium alloys exposed to a dry environment of solid sodium chloride and oxygen, and the present investigation was undertaken to determine whether the austenitic steels would also crack in the same environment.

The greater part of the data presented was obtained using Type 304L (low-carbon 18-10) stainless steel and chemically pure sodium chloride, but Type 304 (18-10), Type 347 (18-12Nb) and Type 430 (17 per cent. chromium) stainless steels, the titanium alloy Ti-6Al-4B, and the salts NaF and NaBr were also included in the study.

Stress-corrosion tests were carried out in the range 200°-1400°F. (95°-760°C.) using constant-load tensile machines, and corrosion spot tests were employed to study the effects of the various salt environments. Susceptibility to corrosion by the vapour phase of sodium chloride was evaluated by sublimation tests, and oxidation-rate data were obtained in continuous-weighing tests. The relative reactivities of Cr, Cr_2O_3, $Cr_{23}C_6$ and Fe with NaCl and O_2 were established in individual corrosion tests. The corrosion products formed during testing were subjected to examination by metallographic, X-ray and wet chemical techniques.

In explanation of the rapid intergranular deterioration which was found to occur in the austenitic steels in the temperature range 1100°-1400°F. (595°-760°C.), the authors propose a mechanism based on the formation of a non-protective Na_2CrO_4-containing scale from the reaction of NaCl and O_2 with Cr_2O_3, Cr and $Cr_{23}C_6$. The reaction rates for Cr_2O_3, Cr and $Cr_{23}C_6$ increased in the order listed, the $Cr_{23}C_6$ reaction occurring very rapidly. Since the last reaction, which is confined to the grain boundaries in austenitic stainless steels, is the most severe, rapid intergranular deterioration of the metal results. Attack was more severe in the 0·08 carbon (Type 304) steel than in the low-carbon (Type 304L) and stabilized (Type 347) stainless steels. Ferritic stainless steels in which the carbide is not confined to the grain boundaries were not found to be susceptible to intergranular corrosion.

Rapid surface oxidation, noted in both austenitic and ferritic stainless steels, was characterized by the formation of a flaky porous and non-adherent scale.

The corrosion process underlying both the intergranular and the uniform attack is considered to involve the migration of oxygen and sodium-chloride vapour to the metal surface. This migration readily occurs, since (*a*) gas phases are involved, and (*b*) the corrosion product is porous. The chemical reactions previously mentioned then take place, producing a non-protective scale containing Na_2CrO_4, and consequently the metal is readily oxidized.

Although it may have a minor influence on the rapid intergranular deterioration of the metal, stress is deemed to have no bearing on the surface attack.

NaBr and NaF salts have a similar effect to that of NaCl, but cause different degrees of surface corrosion. NaF gives rise to surface attack which is many times greater than that resulting from NaCl or NaBr. Surface attack is least with NaBr. All three salts promote rapid intergranular corrosion.

Bibliography on Stress Corrosion

J. DIRIAN, H. CORIOU and L. GRALL: 'Stress Corrosion.' *Commissariat à L'Energie Atomique, Centre d'Etudes Nucléaires de Saclay, Série 'Bibliographies', No. 2,* 1959; 48 pp.+ index.

The *bibliography* covers literature on stress corrosion published over the past 20 years. The 240 references are classified in four sections (relating to (1) Theory and Mechanism, (2) Steels, (3) Light Alloys, (4) Miscellaneous Alloys and Steels), and an indication is given of the scope of each paper covered. The first section is concerned with general studies of the mechanism of stress corrosion; the three others draw attention to theoretical and practical investigations of specific steels or alloys. An author index facilitates reference to the various papers.

Stress-Corrosion Cracking of Austenitic Chromium-Nickel Stainless Steels: A.S.T.M. Report

AMER. SOC. TESTING MATERIALS: 'Report on Stress-Corrosion Cracking of Austenitic Chromium-Nickel Stainless Steels.'
Sponsored jointly by A.S.T.M. Committee A-10 on Iron - Chromium, Iron - Chromium - Nickel and Related Alloys, and N.A.C.E. Committee T-5E on Stress-Corrosion Cracking of Austenitic Stainless Steels.

A.S.T.M. Special Tech. Publn. No. 264, Mar. 1960; 92 pp.

This report contains the results of a comprehensive survey carried out with the object of collecting and assessing information on the type of service in which stress corrosion is most encountered, the grades of steel most often involved, common features of media and service conditions, and the steps being taken to combat cracking.

It was thought that collection and analysis of data relating to actual case histories would establish the magnitude of the problem, and, consequently, in 1956 a questionnaire was distributed to a total of 142 companies and individuals, representing both producers and consumers of stainless steel. Fifty-three replies were received. Of these, 24 companies reported that they had no experience of stress-corrosion cracking; 5 reported that they had had trouble, but possessed no case-history data; the remaining 24 submitted a total of 129 case histories.

As a first step in analyzing the large volume of data obtained, each of the case histories was retabulated in summary form (the summaries are included in an Appendix to the report). The data were classified in five main sections, and the various members of the task group organizing the survey then prepared

a separate review of each section. These reviews comprise *Part* I of the report. Case history data from the United Kingdom, although tabulated in the appendix, were received too late for inclusion in the analyses of Part I.

Part II covers information on current research activities in the field of stress corrosion, obtained both from the questionnaires and from correspondence with all companies, research institutes and universities believed to be conducting such work. It also includes a review of present knowledge of the basic mechanism of the phenomenon in austenitic chromium-nickel steels.

The scope of the papers comprising Parts I and II of the report are indicated below.

Part I. Review of Case Histories

F. L. LAQUE: 'Introductory Summary', pp. 1-3.

The author indicates the scope, and summarizes the results, of the study. He discusses the nature of the problem of stress-corrosion cracking of austenitic stainless steels, identification of stress-corrosion failures, the factors affecting susceptibility to stress corrosion (composition, heat-treatment, structure, surface finish and environment), the time required for cracking, association with general corrosion, and preventive measures (control of stress, cathodic protection, use of inhibitors).

A. W. DANA and W. Z. FRIEND: 'General Information', p. 4.

The authors comment on answers to specific questions included in the questionnaire to obtain information on the extent of the stress-corrosion problem, the application of stress-relieving heat-treatment to prevent cracking, mechanical stress-relieving procedures, and the use of chemical inhibitors or galvanic and cathodic protection.

E. E. DENHARD: 'Equipment, Mode and Location of Fractures and Associated Conditions', pp. 5-6.

In this paper the author has attempted to classify the case histories according to the basic industries with which stress-corrosion cracking is thought to be most frequently associated, the type of equipment involved, time to failure, and location and mode of fracture.

F. K. BLOOM: 'Classification of Materials and Stress Conditions', pp. 7-8.

This section deals with the data classified under 'Materials and Fabrication' and 'Origin of Stress' in the case histories summarized in the appendix. The data analyzed cover the composition, gauge, heat-treatment, hardness and surface finish of the steels, and the origin of stress.

From his evaluation of the information the author concludes that, under appropriate conditions of stress and corrosive environment, any of the austenitic stainless steels are liable to stress-corrosion failure. The grades most frequently appearing in the case histories, A.I.S.I. Types 304 and 316 (18-10 and 18-12Mo), are those most commonly used in the chemical industry, and the incidence of failure in Type 316 does not support the idea sometimes held that this grade is more resistant to cracking than Type 304.

Gauge and finish do not appear to be especially influential factors *per se*, and an attempt to reduce cracking by using stress-relieved and highly-polished tubes was not successful.

Hardness seems to be significant mainly as an indication of whether the material has been severely cold-worked (and therefore contains internal stresses). Residual stresses resulting from fabricating operations are regarded as the most important source of those required to produce cracking.

W. W. BROOKS and M. E. HOLMBERG: 'Classification of Corrosion Conditions', pp. 9-11.

In this section, the data submitted under the heading 'Corroding Media and Service Conditions' and 'Comments' are reviewed in relation to the light they throw on the effects of environment (in particular, the influence of chlorides, fluorides and other halogens), pH, temperature, aeration, and the velocity of the environment.

F. K. BLOOM: 'Steps Taken to Overcome Cracking', pp. 12-14.

Preventive measures are classified in three main categories: substitution of a more resistant material; changes in the corrosive environment (removal of chlorides, minimizing chloride concentration by design modifications, use of coatings to prevent contact with chlorides, lowering the operating temperatures, and treatment of the water); and reduction of residual stresses.

The author's analysis of the data suggests that other materials can sometimes be successfully substituted for austenitic chromium-nickel stainless steel in certain types of equipment. Possible substitutes include carbon steel in mildly corrosive environments, and nickel and nickel-base alloys (such as 'Monel' and 'Inconel') in more agressive environments.

The straight-chromium stainless steels, which appear to be immune to stress cracking in the annealed condition, have received little consideration.

The feasibility of minimizing susceptibility by modification of the environment and by stress-relief is also discussed.

Part II. Present Status of Research Effort

A. W. DANA and W. Z. FRIEND: 'Survey of Current Research Activities', pp. 17-21.

This section contains notes indicating the scope of research being carried out on various aspects of the problem of stress-corrosion cracking by various industrial concerns and Government and university

laboratories in the U.S.A. Foreign investigators believed to be studying stress corrosion of stainless steels are listed.

J. J. HARWOOD: 'The Mechanism of Stress-Corrosion Cracking of Austenitic Stainless Steels', pp. 22–24, 63–67.

In this section, written with the aim of assessing the present state of knowledge of the factors and mechanisms which determine and control the susceptibility of the austenitic stainless steels to stress-corrosion cracking, the author discusses the validity of the various theories proposed in explanation of the phenomenon. Among the factors considered are: the rôle of environment (chloride environments; caustic-bearing water and steam); the influence of temperature; the influence of the composition of the steel; microstructure and thermal-mechanical treatment; surface condition, surface treatment, surface films; stress; the effect of cathodic or anodic currents; and morphology of cracks.

Proposed mechanisms are considered in sections concerned with nucleation or initiation of the crack, crack propagation, electrochemical mechanisms, electrochemical/mechanical mechanisms and stress-sorption cracking mechanisms.

Appendix

'Case History Data from U.S.A. and United Kingdom', pp. 25–61.

The information obtained from the questionnaires are summarized, in tabular form, under the heads: source; general information; material and fabrication method; corroding media and service conditions; origin of stress; comments.

R. M. FULLER, G. T. PAUL and A. J. MARRON: 'Bibliography and Abstracts on Stress-Corrosion Cracking of Stainless Steel', pp. 69–92.

The *bibliography* contains 245 annotated references to literature published from 1935 to 1959 (the items are listed in chronological order).

In addition to stress corrosion proper, the embrittlement of chromium steel is included in the subject matter covered.

Comparative Stress-Corrosion Behaviour of Various Grades of Stainless Steel

P. P. SNOWDEN: 'Comparative Stress-Corrosion Behaviour of Some High-Alloy Steels.'

Jnl. Iron and Steel Inst., 1961, vol. 197, Feb., pp. 136–41.

Although theories have been advanced in explanation of the mechanism of stress corrosion of particular austenitic steels under specific conditions, no extensive comparison has been made of the susceptibility of the various grades available. In the present paper the author reports the behaviour of a number of typical austenitic stainless steels under a variety of corrosive conditions.

The following types of chromium-nickel austenitic steel were selected for study: unstabilized 18–8; low-carbon 18–8; titanium-stabilized 18–8; free-cutting 18–8; 18–8–3Mo; niobium-stabilized 18–9; niobium-stabilized 16–12–1Mo; unstabilized 12–12; titanium-stabilized 18–8–3Mo–3Cu; 25–20. A 20% chromium iron, a 20%Cr–2%Mo–1%Nb ferritic steel, and a 14%Cr–5%Ni–Mo–Cu precipitation-hardenable steel were included in the tests. Tests were carried out at the atmospheric boiling points of solutions of magnesium chloride and sodium chloride, in high-temperature solutions of sodium chloride, sodium hydroxide, and potassium hydroxide, and in high-pressure steam (with specimens contaminated with sodium chloride, sodium hydroxide or potassium hydroxide prior to testing).

On the basis of the corrosion data tabulated, an overall comparison is made of the susceptibility of the various materials to stress corrosion. The author's summary of his conclusions is quoted below:

'Under most chloride and caustic conditions all the 18–8-type steels behaved similarly, with the exception of the 18–8–Ti type, which contained considerable Δ ferrite.

'Increased nickel content, giving a more stable austenite, confers greater resistance to stress-corrosion cracking, and the 12%Cr–12%Ni and 25%Cr–20%Ni types have very good resistance under all the test conditions.

'High Δ ferrite content, as in the 18–8–Ti type material, improves resistance to solutions boiling under atmospheric pressure and to high-temperature caustic solutions, but this behaviour is sometimes erratic, and, with high-temperature chloride solutions, presence of Δ can be detrimental. 18%Cr, 8–12%Ni, 3%Mo steels show little or no improvement over non-molybednum varieties, except perhaps in the superheated steam.

'The ferritic alloys do not suffer stress-corrosion cracking under the conditions employed in these tests, but general attack, or a form of acute pitting, is found under severe conditions. The 20%Cr steel with Mo and Nb shows great improvement over the austenitic steels in chloride environments at high temperatures, but suffers general attack in high-temperature caustic solutions.

'Martensitic alloys (including some not covered in the present work) do not suffer rapid stress-corrosion cracking in steam and high-temperature solutions, although, like the ferritic alloys, general attack occurs under severe conditions. Failures do occur in boiling chloride solutions, depending on hardness and temper condition, but the cracking in this case is often limited to a single crack and may be due to a form of hydrogen embrittlement; this will be discussed more fully in a future paper.

'Finally, then, it may be said that, of the alloys tested, the ferritic alloy with 20%Cr plus Mo and Nb offers the best overall resistance to chloride environments, with the 25%Cr–20%Ni steel giving almost equal performance. Behaviour in caustic environments is much less predictable, but here the higher-nickel alloys show some superiority, and presence of Δ ferrite in an 18–8 steel may be beneficial.'

Feasibility of Preventing Stress Corrosion by Establishing Threshold Values

H. SUSS: 'Practicality of Establishing Threshold Values to Eliminate Stress-Corrosion Failures in Metals and Alloys.'

Corrosion, 1961, vol. 17, Feb., pp. 61t–6t.

Considerable interest has been shown in the feasibility of establishing threshold values for certain parameters, below or above which a material should not be susceptible to stress-corrosion failure in a finite period of time. In this paper, the author reviews the significance of factors which could affect stress-corrosion behaviour, e.g., method of testing, variations normally present in materials conforming to the same specification, slight variations in the test environment or the metallurgical structure of the metal, accelerated corrosive attack (galvanic or crevice), residual stress, and the nature of the corrosive attack.

The review shows that all the significant variables do not act independently, and that there is a complex mutual interaction. It is concluded that the problem of stress corrosion cannot be entirely eliminated at the present time by control of specific parameters to established threshold values. Selection of a more resistant material is the principal approach recommended, but correct design of equipment, reduction of the corrosivity of the environment, reduction of the effective stress, or use of a protective coating can, it is considered, ameliorate the problem.

Influence of Surface Coatings on the Susceptibility of High-Strength and Stainless Steels to Stress Corrosion

E. BAERLECKEN and K. LORENZ: 'Influence of Surface Coatings on the Susceptibility of High-Strength and Stainless Steels to Stress Corrosion.'

Werkstoffe u. Korrosion, 1960, vol. 11, Dec., pp. 744–10.

In this review the authors discuss the factors involved in stress-corrosion cracking of, respectively, austenitic and high-strength steels.

It is shown that the susceptibility of austenitic steels to stress-corrosion cracking is associated with composition: in general ferrite-containing steels are less susceptible, since the ferrite is preferentially attacked and the austenite is cathodically protected. Practical advantage can be taken of the conclusions drawn from these microscale effects by connecting the austenite to a metal which is less noble in the corrosive medium. Application of a chromium coating to austenitic components will result in the formation of a ferritic superficial zone, rich in chromium, which will be anodic to the basis metal in certain electrolytes and provide cathodic protection.

The stress-corrosion cracking of high-strength ferritic steels in acidic hydrogen-sulphide-containing solutions is attributed to the diffusion of atomic hydrogen. Hydrogen pick-up embrittles the material, but can be inhibited by application of anodic current (since the evolution of hydrogen will then take place at the cathode). A surface coating of lead has proved suitable as a cathode, and, since the reaction is electrochemical, any incompleteness or damage of the lead coating will (within certain limits) be without effect.

Caustic Stress Corrosion of 18-12-Nb Steel

E. HOWELLS: 'Caustic Stress Corrosion.'

Corrosion Technology, 1960, vol. 7, Nov., pp. 368–9.

Previous studies have shown that Type 347 18–12–Nb chromium-nickel austenitic steel is susceptible, under certain conditions, to stress-corrosion cracking in aqueous solutions of sodium hydroxide. The tests described in this article were undertaken to obtain quantitative data on these conditions.

Three series of tests were carried out:

1. The influence of the sodium-hydroxide concentration in water and in sodium was studied in tests in which restrained U-bend specimens of Type 347 steel were exposed in small autoclaves. The specimens were annealed at 1020°F. (550°C.) for 15 minutes and water-quenched before stressing.

2. The effect of stress level was investigated in stress-rupture tests on specimens immersed in mixtures of sodium hydroxide and water at 650°F. (345°C.). In all cases, the stress was maintained below the creep-rupture strength to preclude creep cracking.

3. The effects of exposing Type 347 specimens to dilute caustic-water solutions under heat-transfer conditions were studied using miniature boilers. In the tube-to-tube-sheet joint regions, and in the various boiler welds, the fabrication procedure produced stresses at least equal to the yield strength of annealed material. Hydroxides were added to the boiler water on the shell side, and heat was applied by passing pressurized water [at 600°F. (315°C.) and 200 p.s.i.g.] through a single re-entrant tube. With boiler water held at 488°F. (253°C.) and 600 p.s.i.g., a heat-transfer rate of about 100,000 B.Th.U./sq. ft./hour was obtained across the tube. The boiler water contained less than 1 p.p.m. of chlorides, and oxygen was reduced to an undetectable concentration with $NaSO_3$ (tests confirmed the adequacy of these precautions in preventing stress-corrosion cracking).

Other tests were carried out to discover some means of preventing stress corrosion.

From the data presented four conclusions are drawn:

'(1) Caustic stress-corrosion cracks are branching and predominantly transgranular. This is typical of stress corrosion in austenitic stainless steels.

'(2) Threshold stress, if any, is well below yield strength.

'(3) Unlike chloride cracking, caustic stress-corrosion cracking can occur in the absence of oxygen.

'(4) Tests show that less than 0·2 per cent. hydroxide in water may cause cracking in a few hours when concentrating conditions are present. But cracking may be delayed or prevented by adding some disodium phosphate. A minimum addition of one part phosphate to four parts Na+K appears effective.'

Stress-Corrosion Cracking of Stainless Steel De-aerator Trays

N. D. GROVES, L. R. SCHARFSTEIN and C. M. EISENBROWN: 'Chloride Stress-Corrosion Cracking of Stainless-Steel De-aerator Trays.'

Corrosion, 1961, vol. 17, Feb., pp. 53t-4t.

The authors discuss the stress-corrosion failure of stainless steel de-aerator trays, fabricated from A.I.S.I. Type 201* and 329 ('Carpenter 7 Mo')† steels, after a few months' service in a de-aerating feed-water heater. Cracking of the Type 201 trays was very severe.

The feed water [at 62°F. (16°C.), containing 10 p.p.m. chloride, and at a pH of 7·1-7·3] enters a distributing box at the top of the heater, and then flows onto the stacks of trays and down through the unit, to be heated by steam entering at the bottom. The unit is normally maintained at 5 p.s.i. and 227°F. (108°C.).

It is shown that conditions in parts of the heater promote failure by chloride stress-corrosion cracking, and that the service life of austenitic stainless steels is very short. It is considered that the resistance of the Type 329 trays to cracking would be improved by annealing after welding, and Type 430 chromium steel is suggested as a possible material for trays in the upper portion of the heater.

Corrosion-Resistance of Cast Nickel-Molybdenum-Iron Alloys

G. N. FLINT: 'The Corrosion-Resistance of Cast Nickel-Molybdenum-Iron Alloys.'

Metallurgia, 1960, vol. 62, Nov., pp. 195-200.

With the growing complexity of chemical processes, and the continued change from batch to continuous operation, increasingly severe demands are made on materials employed in the construction of chemical plant, and, in many cases, it has become necessary to use materials which have good mechanical properties and a high intrinsic corrosion-resistance even in the absence of a protective surface film. Among the materials falling within this classification are the nickel-molybdenum-iron alloys which, even at high temperatures, exhibit outstanding resistance to corrosion by hydrochloric, sulphuric and phosphoric acids (and find extensive use in critical applications such as pump and valve parts, heat exchangers, agitators and thermocouple sheaths). This article reports data demonstrating the influence of composition and heat-treatment on the hardness, and resistance to corrosion by strongly acid media, of cast nickel-molybdenum and nickel-molybdenum-iron alloys.

The alloys, containing 0-29·2 per cent. iron and up to 31 per cent. molybdenum (the three main ternary series tested contained 20, 25 and 30 per cent. molybdenum), were heated at 1180°C. for 5 hours. Test specimens of the alloys containing 20 per cent. or more molybdenum were re-heated at 800°C. for 5 hours and air-cooled, and two 30 per cent. molybdenum alloys (containing, respectively, 0·76 and 6·2 per cent. iron) were re-heated at 700°C. for periods of up to 300 hours. The hardness of the low-iron alloy was also determined after treatment at various temperatures between 675° and 860°C.

Corrosion-resistance was evaluated in relation to the following media: aerated 10% H_2SO_4 at 50°C. (3-day test); aerated or nitrogenated 5% HCl at 50°C. (3-day test); boiling 5% and 10% HCl (1-day test). A specimen of wrought nickel was included in some of the corrosion tests for reference purposes. Corrosion rates were determined, and sections of the corroded alloys were subjected to micro-examination. The potentials of the alloys, when corroding freely in unagitated 5% HCl at room temperature, were established, and anode-polarization measurements were made on nickel and nickel-molybdenum alloys in an aerated 5% HCl solution at 30°C.

The data presented are discussed in terms of the influence of composition on corrosion-resistance, and the influence of heat-treatment on microstructure, corrosion-resistance and hardness.

The results of the investigation demonstrate that, for most applications, an alloy containing molybdenum 26-30, iron 4-7, per cent., balance substantially nickel (i.e., the composition of a number of commercial alloys) exhibits optimum corrosion-resistance and is associated with the economy in production resulting from the use of ferro-molybdenum. Subsequent experience has indicated that the corrosion-resistance of the alloy is normally equally satisfactory in the as-cast condition and after the heat-treatment at 1180°C. employed in the present tests.

In discussing the effects of heat-treatment on composition, microstructure, corrosion-resistance and hardness, the author draws the conclusion that the addition of iron to nickel-molybdenum alloys suppresses precipitation of β and favours that of γ; hence it impairs the hardening response of nickel-molybdenum-iron alloys on ageing. Since, moreover, γ is richer in molybdenum than is the matrix, its precipitation is more likely to lower corrosion-resistance, by molybdenum depletion of the matrix, than precipitation of β, which has approximately the same molybdenum content as the matrix.

For applications requiring resistance to both abrasion and corrosion, an alloy containing 30 per cent. molybdenum and less than 1 per cent. iron is therefore preferred, since in the aged condition it has much greater hardness and better corrosion-resistance than that containing 4-7 per cent. iron.

* Composition: carbon 0·09, chromium 18·18, manganese 7·22, nickel 5·25, molybdeum 0·18, per cent.

† Composition: carbon 0·08, chromium 27·37, nickel 4·21, molybdenum 1·68, per cent.

Influence of Moisture on the Resistance of Materials to Corrosion by Bromine

D. E. LAKE and A. A. GUNKLER: 'Moisture: Key to Bromine Corrosion.'

Chemical Engineering, 1960, vol. 67, Oct. 31, pp. 136, 138.

When completely dry, bromine is not corrosive to many non-ferrous metals, including nickel, copper and magnesium (it is highly reactive, wet or dry, with aluminium). Commercial bromine is shipped with a water content below 20–30 p.p.m., but its affinity for moisture may render it highly corrosive, through pick-up of additional water, if incorrectly handled: at room temperature the water content of bromine could, in contact with air of 20 per cent. relative humidity, reach 70 p.p.m., a value which, in the case of exposure to air having 80 per cent. relative humidity, could rise to 300 p.p.m. To determine the influence of moisture content on the corrosion-resistance of various metals, laboratory tests were run under conditions in which humidity was controlled with baths of sulphuric acid.

Tests were carried out on nickel, 'Monel', aluminium-bronze, Muntz metal, chemical lead, Ounce metal (copper 85, zinc 5, lead 5, tin 5, per cent.), copper, carbon steel and Type 316 18–12–Mo stainless steel. Three samples of each material were exposed: one totally immersed, one semi-immersed, and the third exposed to bromine vapour. The corrosion rates of the more satisfactory materials are reproduced below (in in./year $\times 10^{-3}$).

Material and Exposure Conditions	Water in Bromine	
	7 p.p.m.	57 p.p.m.
'Monel', liquid Br_2 ..	0·10	0·46
'Monel', vapour	0·04	1·32
Nickel, liquid	0·07	0·20
Nickel, vapour	0·05	0·83
Aluminium bronze, liquid	0·27	0·18
Aluminium bronze, vapour	2·86	66·80
Muntz Metal, liquid ..	0·35	0·80
Muntz Metal, vapour ..	0·79	1·39
Chem. lead, liquid.. ..	0·53	2·28
Chem. lead, vapour ..	1·67	3·64
Ounce metal, liquid ..	0·46	1·76
Ounce metal, vapour ..	0·05	0·45

In other sections of the article reference is made to tests indicating that (1) in the case of nickel specimens, initial corrosion rates are not excessive, and (2) impure bromine can have a more corrosive effect than commercially pure bromine. Summarizing their conclusions, the authors emphasize the desirability of maintaining the water content of bromine in the 20–30 p.p.m. range.

Influence of Moisture on the Resistance of Materials to Chlorohydrocarbons

G. P. GLADIS: 'Effects of Moisture on Corrosion in Petrochemical Environments.'

Chemical Engineering Progress, 1960, vol. 56, Oct., pp. 43–51.

Each new process and product developed by the petrochemical industry confronts the engineer with the problem of selecting suitable materials of construction for process equipment. This review is 'limited to the examination of one particular source of corrosion, found in many petrochemical operations and often overlooked as a cause of considerable equipment deterioration and product contamination': the presence of water. Large amounts of chlorinated hydrocarbons (chlorohydrocarbons) are produced from natural gas or petroleum gases, for use either as starting materials and intermediates for further petrochemical processing stages, or as finished products. The major part of the paper is concerned with the resistance of materials of construction to corrosion in wet chlorohydrocarbon media.

In the initial sections of the review, the author discusses the effects of moisture on the corrosiveness of chlorinated compounds, and presents data (derived from laboratory and plant corrosion tests) demonstrating the behaviour of various metals and alloys in wet chlorohydrocarbon media. He then surveys the relative resistance of steel and cast iron, copper and copper-base alloys, high-nickel alloys, aluminium and titanium to the following wet media: chlorohydrocarbons, chlorine, ammonia, sulphur dioxide. The final section is made up of practical hints on ways of extending the life of equipment processing wet corrosive media.

The corrosion data tabulated relate to copper, aluminium, brass, lead, zinc, 'Monel', 'A' nickel, cast iron, 'Ni-Resist', 'Inconel', carbon steel, Types 302, 304, 316 and 317 chromium-nickel stainless steels. 'Hastelloy B', 'Hastelloy C', 'Carpenter 20' and 'Ni-O-Nel'. The corrosion tests were carried out in the following wet environments (though not all the materials were included in any one test series): in carbon tetrachloride; in steam distillation and rectification of crude trichloroethylene; in vapour containing 87·5% CCl_4, 12% H_2O, 0·4% Cl_2, 0·1% HCl; in monochlorobenzene containing traces of water and phenol; in activated-carbon solvent-recovery systems; in gases containing 2–5% or 13–17% SO_2 and saturated with water vapour; in fumes containing 5% SO_3, 8% CO_2, 11% O_2 and SO_3 mist from calcining operations; and in an adsorption tower with SO_2 gas saturated with water vapour.

Low-Temperature Mechanical Properties of Structural Materials: Compilation of Published Data

See abstract on p. 54.

Design and Operation of Ethanolamine Gas-Scrubbing Systems

G. D. HALL and L. D. POLDERMAN: 'Design and Operating Tips for Ethanolamine Gas-Scrubbing Systems.'

Chemical Engineering Progress, 1960, vol. 56, Oct., pp. 52–8.

The aqueous, or Girbitol, process is the method most widely used in North America for removing carbon dioxide and/or hydrogen sulphide from refinery gases and liquids, natural gases, stack gases and chemical-plant synthesis gases. The Girbitol process has been in use since the 1930's, and over the years much has been learnt about plant design and methods of minimizing operating problems, so that to-day most plants operate relatively trouble-free. Operating problems are, however, sometimes encountered, and those most common are discussed in the present paper.

Sections on degradation of the ethanolamines, and causes and control of foaming, are followed by notes on the design of monoethanolamine reclaimers, sludge-accumulation problems, failure to sweeten effluent gas, and corrosion problems. The factors of importance in minimizing corrosion of the materials of construction are dealt with in some detail, and are exemplified in the authors' discussion of four case histories of plant corrosion. Design and operating procedures which contribute to prevention of corrosion are outlined.

As a guide to the selection of suitable materials of construction, the authors comment:

'Normally, carbon-steel equipment is satisfactory in both monoethanolamine and diethanolamine service, but stress-relieving of all major process equipment is important. Where experience indicates an unsatisfactory short life for carbon-steel tubes, and plant operating practices cannot be revised to reduce the corrosiveness of the environment, the use of corrosion-resistant alloys is indicated. Experience indicates that Type 316 (18–12–Mo) stainless steel is most satisfactory, while Type 304 (18–10) stainless gives good service at lower cost in most cases. It should be noted that there are some cases on record where Type 304 failed rapidly. It is desirable to expose test tubes of both Types 304 and 316 in a bundle before selecting either material. The tubing should be purchased in the annealed condition. Either seamless or welded tubing may be used, the latter being somewhat less expensive.'

Corrosion-Erosion of Sensitized Stainless Steel in a Thorium-Dioxide/Uranium-Trioxide Slurry

D. C. VREELAND: 'Corrosion-Erosion of Sensitized A.I.S.I. Type 304 Stainless Steel in a Thorium Dioxide-Uranium Trioxide Slurry.'

Corrosion, 1961, vol. 17, Jan., pp. 21t–4t.

As part of a programme designed to throw light on the corrosion-erosion behaviour of materials in a thorium-dioxide/uranium-trioxide slurry under consideration for use as fuel, moderator and coolant in a nuclear-reactor power plant, sensitized sheet specimens of A.I.S.I. Type 304 18–10 chromium-nickel stainless steel were installed, for evaluation, in dynamic-testing loops. The data obtained from the test programme are reported in this paper.

Oxide which formed on the specimens during sensitization was removed either by grinding or by pickling in HNO_3/HF solution. Sensitized and pickled specimens were subjected to variable-velocity conditions in Loop A; specimens subjected to constant-velocity conditions were exposed in Loop B. For comparative purposes, coupons of Type 304 steel in the 'as-received' and in the pre-filmed conditions were included in Loop B. Some tests were also carried out on Type 347 18–10Nb steel. Conditions during loop operation were as follows:

Loop A

Mean slurry concentration: 320 gm. ThO_2 and 11·7 gm. UO_3 per liter.
Temperature: 580°F. (305°C.).
Pressure: 1650 p.s.i.g. (115·5 kg./cm.^{2} above atmospheric pressure).
Oxygen: 770 p.p.m.
Slurry velocity past coupons: 25–88 ft./sec. (7·5–26·5 m./sec.).
Time of test: 1000 hours.

Loop B

Mean slurry concentration: 330 gm. ThO_2 and 12·2 gm. UO_3 per liter.
Temperature: 580°F. (305°C.).
Pressure: 1700 p.s.i.g. (119 kg./cm.^{2} above atmospheric pressure).
Oxygen: none.
50 p.s.i. N_2 overpressurization.
Slurry velocity past coupons: 28 ft./sec. (8·5 m./sec.).
Time of test: 59, 257, 330, 494 or 1170 hours.

The conclusions drawn from the data are summarized by the author as follows:

'1. Sensitized non-pickled Type 304 stainless steel was attacked at approximately the same rate as was as-received Type 304 in test-loop systems at a velocity of approximately 28 ft./sec. Sensitized non-pickled steel was not attacked intergranularly in the loops.

'2. Type 304 stainless steel, sensitized and pickled in HNO_3–HF, was attacked at appreciably higher rates than was as-received Type 304 or sensitized, non-pickled Type 304 in test-loop systems at a velocity of approximately 28 ft./sec.; however, when the layer of metal attacked by the pickling solution was removed by the action of the slurry, the attack diminished.

'3. At higher velocities, somewhat over 28 ft./sec., sensitized and pickled Type 304 stainless steel was continuously attacked at significantly higher rates than was as-received Type 304 or Type 347. This is believed to be due to the rough surface that a pickled specimen presents to the flowing slurry. The rough surface tends to form stronger eddies as the

slurry flows at higher velocities, and thus the erosion of the specimen is increased.

'4. To minimize corrosion-erosion of sensitized Type 304 components in high-velocity slurry under loop-operating conditions, pickling after sensitization and prior to test-loop exposure should be avoided. In practice it was found unnecessary to pickle loop components, since any heat-tint resulting from welding did not interfere with loop operation. After construction, loops usually were cleaned by being operated for short periods while containing a detergent solution. This procedure removed grease and other foreign material from the system.

'5. As-welded Type 304 would not be expected to be susceptible to intergranular corrosion by the environment studied when the heat tint caused by welding is not removed by a pickling operation.'

Low-Temperature Properties of Ferrous Materials

See abstract on p. 54.

Resistance of Nickel-containing Materials to Liquid Fluorine and Liquid Oxygen

F. W. FINK and E. L. WHITE: 'Corrosion Effects of Liquid Fluorine and Liquid Oxygen on Materials of Construction.'

Corrosion, 1961, vol. 17, Feb., pp. 58t–60t.

This paper was presented at the 16th Annual Conference of the National Association of Corrosion Engineers, 1960, and has been abstracted, from the preprint then available, in *Nickel Bulletin*, 1960, vol. 33, No. 7, p. 180.

Low-Temperature Properties of Cold-Worked or Welded Stainless-Steel Sheet

J. F. WATSON: 'Mechanical Properties of High-strength 301 Stainless-Steel Sheet at 70, −320 and −423°F. in the Base Metal and Welded Joint Configurations.'

Advances in Cryogenic Engineering, vol. 5, pp. 406–20; disc., p. 420.

Distributed by Plenum Press, Inc., New York, 1960.

In the investigation reported, the mechanical properties of Type 301 18–8 chromium-nickel steel were determined using sheet specimens which had been cold-worked 42, 62 or 78 per cent. The tensile properties of heliarc butt-welded joints in similar specimens were also established.

Toughness was studied by means of notch-tensile tests, and the notch-to-unnotch tensile ratio subsequently calculated was taken as a criterion of resistance to brittle fracture.

The low-temperature properties of the cold-rolled stainless steel were found to depend on both the original austenitic structure and on martensite transformation induced by combinations of low temperature and plastic strain. Reductions in tensile strength and elongation in the range −320° to −423°F. (−196° to −253°C.), indicative of the occurrence of the austenite-to-martensite transformation, were accompanied by a decrease in the notch-to-unnotch tensile ratios. The solid-state transformation of austenite to martensite was confirmed and measured both by magnetic and X-ray-diffraction techniques. The data demonstrate that a reduction of about 60 per cent. was optimum for achievement of high notch-to-unnotch ratios. Reductions above this amount resulted in excessive low-temperature embrittlement (due to the presence of large amounts of martensite formed by cold work at room temperature), while lesser amounts of cold work did not introduce sufficient defects into the lattice to impede the austenite-to-martensite reaction resulting from low temperature and high strain during deformation.

Heliarc butt-welded joints exhibited a much larger percentage increase in tensile strength between 70° and −320°F. (21° and −196°C.) than the cold-worked specimens. Weldments also exhibited embrittlement in the range −320° to −423°F. (−196° to −253°C.), reflected in decreases in tensile strength, accompanied by wide scatter in the test data.

Preferential Corrosion of Stabilized Stainless-Steel Welds

C. L. ANGERMAN and P. M. KRANZLEIN: 'Preferential Corrosion of Stabilized Stainless-Steel Welds.'

Amer. Soc. Metals, 1960, *Preprint No.* 208.
Trans. Amer. Soc. Metals, 1961, vol. 53, pp. 433–46.

Previous studies of the corrosion of welded A.I.S.I. Type 309SCb (niobium-stabilized 23–14 chromium-nickel) stainless steel in a 3·0M HNO_3/0·075M HF solution had shown the weld metal to suffer severe interdendritic corrosion. The investigation reported was undertaken to study the mechanism of this type of attack.

Although the major portion of the work was carried out on welds produced in Type 309SCb, a brief study was made of the corrosion characteristics of Type 347 and 321 (18–12Nb and 18–11Ti) stainless-steel welds, and comparative tests were made on wrought specimens of Type 304 (unstabilized 18–8) steel. Corrosion mechanisms were studied by subjecting the weld specimens (in the as-welded or heat-treated condition) to electron-microscopical examination before and after corrosion tests in two solutions: boiling 3·0M HNO_3/0·075M HF, and boiling 65 per cent HNO_3. The carbides present in the weld metal were identified by electron diffraction.

In both solutions, weldments in the stabilized steels were found to corrode by preferential attack on particles (identified as NbC in Types 309Cb and 347, and TiC in Type 321) located along grain boundaries. This mechanism contrasts with that involved in the attack on the chromium-depleted zone adjacent to the chromium-carbide precipitate in such unstabilized stainless steels as Type 304.

General corrosion was observed in welds produced using filler rods of the same composition as the basis metal, whereas welds in which Type 308L filler rods were employed suffered corrosion similar to knifeline attack (characterized by corrosion of the

NbC or TiC particles that had formed at grain boundaries in the zone between the weld metal and the basis metal).

Sigma phase, which was present in welds heated at 1650°F. (900°C.) for times up to 20 hours, but was not observed in as-welded metal, did not appreciably change the corrosion rate of either the weld metal or the basis metal.

A brief survey of the literature revealed no other examples of severe corrosion due to attack on an interdendritic network of NbC or TiC. Both weld corrosion and knifeline corrosion of the stabilized steels have been thought to be caused by the formation of either $Cr_{23}C_6$ or sigma phase.

Combined Method for Analysis of High-Alloy Irons and Steels

W. B. SOBERS: 'Combined Method of Analysis for High-Alloy Iron and Steel.'

Foundry, 1960, vol. 88, Sept., pp. 110–13.

The author gives details of an analytical procedure (involving the use of dilution techniques) applicable to the determination of the following elements in a single sample of high-alloy iron or steel: silicon, manganese, phosphorus, chromium, nickel, molybdenum, copper. The technique obviates the need for weighing and dissolving several samples, and thus saves time, chemicals and laboratory space.

The aliquot samples for the various determinations are taken from a master solution. The preparation of this master solution, and the gravimetric, colorimetric or volumetric procedures used in determining each of the seven elements, are outlined.

Chemical Milling

See abstract on p. 55.

ANALYSIS

Combined Method for Analysis of High-Alloy Irons and Steels

See abstract on this page.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Trade Marks*.

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